REPORT 1058

INFLUENCE OF CHEMICAL COMPOSITION ON RUPTURE PROPERTIES AT 1200° F OF FORGED CHROMIUM-COBALT-NICKEL-IRON BASE ALLOYS IN SOLUTION-TREATED AND AGED CONDITION [1]

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SUMMARY

The influence of systematic variations of chemical composition on rupture properties at 1200° F was determined for 62 modifications of a basic alloy containing 20 percent chromium, 20 percent nickel, 20 percent cobalt, 3 percent molybdenum, 2 percent tungsten, 1 percent columbium, 0.15 percent carbon, 1.7 percent manganese, 0.5 percent silicon, 0.12 percent nitrogen, and the balance iron. These modifications included individual variations of each of 10 elements present and simultaneous variations of molybdenum, tungsten, and columbium. Laboratory induction furnace heats were hot-forged to round bar stock, solution-treated at 2200° F, and aged at 1400° F. The melting and fabrication conditions were carefully controlled in order to minimize all variable effects on properties except chemical composition.

For the limited number of composition variables studied the range in 100-hour rupture strengths was from 26,000 to 52,000 psi. Major strengthening effects resulted from additions of molybdenum, tungsten, and columbium, individually or simultaneously. The lowest-strength alloy contained none of these elements. Chromium also had a major strengthening influence. However, no alloy was obtained which had properties which were outstanding compared with those of the basic analysis.

Carbon (varied from 0.08 to 0.60 percent), nitrogen (0.08 to 0.18 percent), manganese (0.30 to 2.5 percent), nickel (10 to 20 percent), cobalt (20 to 32 percent), and columbium (2 to 4 percent) had little influence on rupture properties. Nitrogen (0.004 to 0.08 percent), chromium (10 to 30 percent), nickel (0 to 10 percent), cobalt (0 to 20 percent), molybdenum (0 to 4 percent), tungsten (0 to 4 percent), and columbium (0 to 1 percent) increased strength appreciably. Silicon (0.5 to 1.6 percent) and nickel (20 to 30 percent) had a weakening influence. Columbium had a marked influence on increasing total elongation to fracture.

Rupture strengths varied directly with a measure of the resistance to creep of the alloys with total elongation to fracture as a parameter. This indicated that rupture strengths were a function of the effect of the composition modifications on both the inherent creep resistance and the amount of deformation the alloys would tolerate before fracture.

Interpretation of the results on the basis of microstructural studies indicated that molybdenum and tungsten improved creep resistance by entering solid solution. Nickel and cobalt, two elements forming part of the matrix solid solution, appeared to improve strength by increasing the solubility of molybdenum and tungsten. Chromium improved creep resistance, at least in part, as a result of an aging reaction. Columbium did not appreciably affect creep resistance but improved rupture strength by increasing the elongation to fracture.

Information is presented which indicates that melting and hot-working conditions play an important role in high-temperature properties of alloys of the type investigated.

INTRODUCTION

This investigation had as its object a study of the influence of systematic variations of chemical composition on the 1200° F rupture properties of forged alloys in the solution-treated and aged condition in which the composition was varied from the following basic analysis:

Chemical composition (percent)			
Carbon, C	0.15	Cobalt, Co	20
Manganese, Mn	1.7	Molybdenum, Mo	3
Silicon, Si	0.5	Tungsten, W	2
Chromium, Cr	20	Columbium, Cb	1
Nickel, Ni	20	Nitrogen, N	0.12
		Iron, Fe	32

Sixty-two modifications of this alloy were studied in which each of 10 elements was systematically varied individually and Mo, W, and Cb were varied simultaneously.

The investigation was prompted by the fact that at present there are no published data which make possible a correlation between systematic variations in chemical composition and the high-temperature properties of forged Cr-Ni-Co-Fe-Mo-W-Cb alloys of the type investigated. Past research has shown that close control over processing of alloys is necessary to reproduce high-temperature properties between different heats of the same analysis. Lack of such control of processing variables has resulted in the failure of attempts to correlate the published high-temperature data on these types of alloys. Such correlations are needed to provide a basis for establishing optimum chemical compositions for heat-resistant alloys, for reducing the required alloy content while retaining worth-while properties, and for determining the fundamental mechanisms by which alloying elements influence properties at high temperatures.

As an initial approach to the solution of a large problem, the investigation was necessarily limited in scope. The

[1] Supersedes NACA TN 2449, "Investigation of Influence of Chemical Composition on Forged Modified Low-Carbon N-155 Alloys in Solution-Treated and Aged Condition as Related to Rupture Properties at 1200° F" by E. E. Reynolds, J. W. Freeman, and A. E. White, 1951.

number of compositions studied, the use of only one condition of processing, the evaluation of high-temperature characteristics only by rupture properties for 100 hours at 1200° F, and the use of only microstructural and hardness data to provide interpretation of results were the major limitations placed on the investigation.

This work was conducted at the University of Michigan under the sponsorship and with the financial assistance of the National Advisory Committee for Aeronautics.

EXPERIMENTAL PROCEDURE

The basic analysis selected for study of the influence of systematic variations in chemical composition on rupture properties at 1200° F was that of a commercial alloy which has the following nominal composition:

Chemical composition (percent)			
Carbon	0.15	Cobalt	20
Manganese	1.7	Molybdenum	3
Silicon	0.5	Tungsten	2
Chromium	20	Columbium	1
Nickel	20	Nitrogen	0.12
		Iron	32

The influence of composition was evaluated on 62 modifications of this alloy with the following composition variables:

(1) Individual variations of the elements in the basic analysis:

C, percent	0.08, 0.40, 0.60
Mn, percent	0, 0.30, 0.50, 1.0, 2.5
Si, percent	1.2, 1.6
Cr, percent	10, 30
Ni, percent	0, 10, 30
Co, percent	0, 10, 32
Mo, percent	0, 1, 2, 5, 7
W, percent	0, 1, 5, 7
Cb, percent	0, 2, 4, 6
N, percent	0.004, 0.08, 0.18

(2) Simultaneous variations, in steps of 2 percent, of Mo, W, and Cb from 0 to 4 percent

In all cases Fe variations compensated for the variations of total alloy content.

The first step in the investigation was the development of melting, forging, and heat-treatment procedures which would minimize all variable effects on properties except chemical composition. This work was done on the basic alloy with the reproducibility of 1200° F rupture properties from heat to heat being the main criterion for final adoption of preparation procedures. Six heats were prepared to develop melting practice. Three additional heats were prepared using varying deoxidation practices. The forging procedure was developed on six additional heats of the basic alloy.

The work on procedure development and detailed descriptions of the final procedures adopted are summarized in the subsequent section "Preparation of Experimental Alloys." Briefly these consisted of melting the alloys as 9-pound induction-furnace heats, hot-forging the ingots between 2200° and 1800° F to bar stock, heat-treating at 2200° F for 1 hour, and water-quenching followed by aging at 1400° F for 24 hours.

All alloys were chemically analyzed for the modified element while complete analyses were made only on spot heats within a group after it was established that melting practice was yielding the desired compositions.

Metallographic examinations were made of all the alloys in the as-cast, hot-forged, solution-treated, aged, and rupture-tested conditions. Vickers hardness tests were made on bar stock in the solution-treated and the aged conditions.

The high-temperature load-carrying ability of the alloys was evaluated by means of stress-rupture properties at 1200° F. Rupture test specimens, machined from the heat-treated bar stock, were 0.250 inch in diameter with a 1-inch gage length. The stress-rupture tests were made in individual stationary units with the load applied by a simple beam acting through a system of knife edges. At least two and usually three or four tests at various stresses were made on each alloy and were of sufficient duration to establish the 100-hour rupture strengths and to permit at least an estimate of the 1000-hour strengths. Time-elongation data were taken during the rupture tests by the drop-of-the-beam method and also, in the case of many of the longer time tests, by means of modified Martens type extensometers with a sensitivity of 0.00005 inch per inch. There was good agreement between curves from the two types of deformation measurements in all cases where both types were used on the same tests.

PREPARATION OF EXPERIMENTAL ALLOYS

This section discusses the development of techniques for melting, forging, and heat-treating, describes the procedures finally adopted, presents the observed behavior of the alloys during processing, and shows the reproducibility of rupture properties of the basic alloy resulting from the processing procedures used.

MELTING

Preliminary melting experiments.—Six heats were prepared originally to develop and standardize melting practice, particularly to obtain control over final composition from charge calculations and reproducibility from heat to heat. Actual analyses of these heats are given in table I. It was found necessary to make certain minor corrections in the charged materials and the next five heats of the basic alloy had reasonably consistent compositions.

Melting procedure.—The alloys were melted in a 12-pound-capacity induction furnace as 9-pound heats. The heats were poured into 9-inch-long tapered (1⅝- to 1¼-in.-sq.) cast-iron ingot molds with 2⅛-inch-square hot tops. The life of the magnesia melting crucible was from 10 to 12 heats.

A typical melting charge and schedule are given in table II. This table shows the various ferroalloys used to make up the charges. The Fe, Cr, Ni, and Co charge was first melted down; Mn and Si were added, followed by Mo, W, and Cb. The heat was then deoxidized with 15 grams of calcium-silicon alloy, power turned off, bath temperature taken with both a Leeds and Northrup optical pyrometer and a platinum, platinum-rhodium immersion thermocouple, and the metal poured. The calibration of the immersion thermocouple was checked periodically.

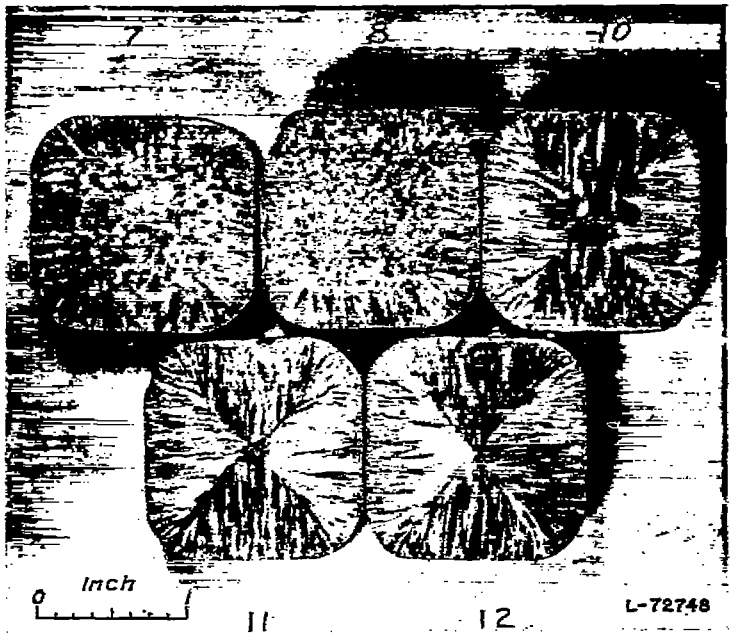
Immediately after pouring, a Chromel-Alumel thermocouple was immersed into the molten metal in the hot top and the cooling curve determined by readings at 5-second intervals on a Leeds and Northrup portable potentiometer.

In a subsequent study of the effect of deoxidation on properties, three heats of the basic alloy were prepared using deoxidation practices varying from the normal use of 15 grams of calcium-silicon alloy. The three variations were: No deoxidation (alloy 74); 15 grams of zirconium-silicon-iron alloy (75); and melting under a lime-fluorspar slag (76).

Melting characteristics.—Table I gives the chemical compositions of the experimental alloys. The intended modifications from the basic alloy are given along with actual values where chemical analyses were made. In general, the actual analyses were consistently close to the desired composition.

Table III gives bath temperatures just prior to pouring the alloys. There was a fair agreement and no consistent difference between the optical pyrometer and the immersion thermocouple readings. Cleanness of the bath surface was found to be of importance in the pyrometer readings. The agreement between the two methods in so many cases indicates that the temperatures given are reliable.

A macrographic section of the ingot cross section was taken from each heat from just below the hot top to determine if pipe or porosity existed. All of the heats actually tested were sound. The ingot grain structure was well-defined on these sections, a typical example of which is shown in figure 1 for several heats. In general the various ingot macrostructures reflected the relative pouring temperatures rather than variations in chemical composition. The higher the pouring temperature, the greater was the total area covered by the columnar grains and, the greater the size, the less the total area covered by the equiaxed grains at the center.



Alloy	Approximate pouring temperature (°F)
7	2570
8	2560
10	2660
11	2650
12	2785

FIGURE 1.—Typical macrostructures of ingots. Etchant, Marble's reagent.

The cooling curves obtained on the metal in the ingot hot tops were similar in that, in all cases when readings were begun early enough to determine it, they had a definite halt in temperature within what seemed to be the solidification range for the alloys. Usually these halts were preceded on the curve by what appeared to be an undercooling effect. The time of the constant temperature halt varied for the alloys from 10 to 60 seconds. After these halts the cooling curves were smooth with a gradual decrease in cooling rate as the temperature decreased. The last column in table III contains the temperatures of halts in the cooling curves during solidification. In cases where temperature readings were not started early enough the halt temperature is indicated as being greater than that at which the cooling curve was started.

The over-all temperature range of the halts was from about 2420° to 2610° F and they varied consistently with systematic chemical-composition variations. Alloying additions tended to lower the temperature of the halt, with Cb having the greatest effect. The exact physical significance of the temperature halts, beyond an indication of the temperatures at which the major portion of the alloy solidified, is uncertain.

FORGING

Preliminary forging experiments.—There was available for the forging work a Nazel air forging hammer (size 3, type B) which is approximately equivalent to a 400-pound steam hammer. This hammer was fitted with 3½- by 8-inch flat dies.

Preliminary forging to determine the forging temperature range for the alloys was done on ⅞-inch-square bar stock of the basic alloy, reducing it to 0.43-inch squares from 2100°, 2200°, 2300°, and 2400° F. The plasticity increased with temperature, but burning, which resulted in forging cracks, occurred at 2400° F. Further experiments on a 0.55-percent-C heat (alloy 5) indicated that burning could occur at 2300° F in this alloy. Since the alloy was quite difficult to forge with an initial temperature of 2100° F it was decided to use 2200° F as the initial forging temperature. Subsequent work on the heats with variable composition showed that all the alloys were forgeable from this temperature, the forgeability, however, varying with the composition.

Finishing forging temperatures were kept at 1800° F or above as judged by color, in order to minimize hot-cold-work, which is known to influence the properties of these alloys.

Several ingots were forged to ½-inch-square bars between the flat dies, a reduction of approximately 90 percent. Microstructural examination of these forged bars showed a nonuniformity of grain size in the cross section. A diagonal "X," distinctly visible on visual examination of the etched cross section, was present in all the bars. The grains were appreciably finer along these diagonal planes than in the triangles, under the flat surfaces of the bar, formed between these planes.

A number of different methods of forging were tried in order to determine the cause of and in an effort to eliminate this grain size nonuniformity. The methods used and their results were:

(1) Forging at a 45° angle to the original square of the ingot resulted in the same diagonal "X" from the corners of the final square indicating that the ingot structure bore no relation to this characteristic.

(2) Upset forging prior to final reduction to a square showed no advantage of the added working on the structural uniformity.

(3) Finishing as an octagon between flat dies yielded areas of fine grains originating at all eight corners of the bar.

(4) Forging wholly between a flat top die and bottom "V" dies to an octagon appeared to abuse the metal more than any other method used. It was difficult to draw the metal out, the cross section merely being changed back and forth. This tended to open up the center of the bar.

(5) Forging to approximately 1 inch square and finishing to ½ inch round between hand swages in four steps gave the best structural uniformity. A narrow band of fine grains was sometimes present near the surfaces of the round bars but the over-all grain size was quite uniform.

The above experiments showed that the forging "X" in the square bars was the result of the relatively greater amount of hot-work along the diagonal planes. This was further verified by the fact that occasionally in forging the square bars diagonal cracks would form completely through the bars.

As a result of the forging experiments it was decided to fit the hammer with a set of dies with swaging impressions. Subsequent experiments using this method proved satisfactory so it was adopted as the standard forging procedure for the experimental alloys.

Forging procedure.—The general arrangement of the 400-pound-capacity air forging hammer and the heating furnace is shown in figure 2. A picture of the forging operation in swaging dies is shown in figure 3. Figure 4 shows a bar which was forged to indicate the steps in reduction from the ingot to the final round bar. A typical forging record is presented in table IV.

The temperature of the furnace was controlled by an automatic temperature controller through a platinum, platinum-rhodium thermocouple. The temperature of the forging stock was measured by a Chromel-Alumel thermocouple attached to a small bar placed next to the stock.

The cast ingots were ground to remove all surface defects prior to forging. The ingots were preheated to approximately 1400° F, then placed just inside the door in the coolest region of the furnace, and finally moved to the center of the furnace at 2200° F.

Generally half of an ingot was forged at a time. After holding at least 30 minutes at 2200° F the ingot was forged from approximately 1.4 inches square to slightly over 1 inch square between flat dies with from two to four reheats. A number of the blows in the flat dies were on the corners of the bars to prevent corner cracks and to prepare the piece for the first swage. The bar was reheated and forged to approximately 0.95 inch round in the first swage with from one to three reheats. The second swage reduced the bar to approximately 0.75 inch round with two to four reheats. The forged bar was then cut off the unforged half of the ingot and recharged to the furnace. The third swage reduced the bar to approximately 0.58 inch round with three to six reheats. The bar was then cut in two equal pieces for the final swaging. The last swage finished these bars to 0.40 to 0.50 inch round with from 5 to 10 reheats. The number of blows from the hammer were counted and tabulated. The number of reheats and blows varied depending on the ease of forging of the alloy and the size of the stock. Total reduction of area during forging was approximately 90 percent

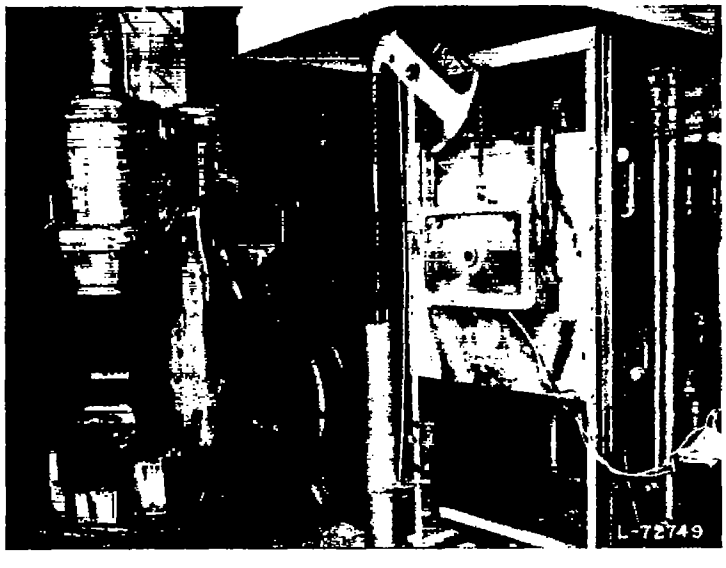


FIGURE 2.—Arrangement of forging hammer and furnace.



FIGURE 3.—Forging experimental alloy in swaging dies.

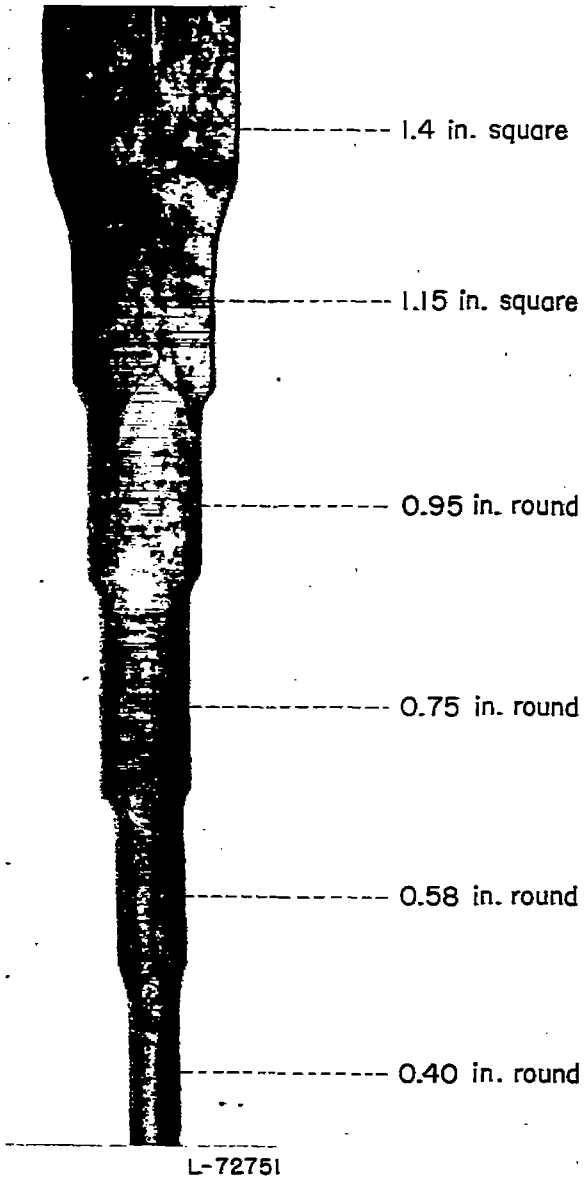


FIGURE 4.—Forged bar showing reduction steps used in swaging experimental alloys from original ingot to final round bar stock.

Reduction in the individual swages was done in steps; that is, after the first reheat only a portion of the bar was reduced to the swage diameter, after the second reheat another portion of the bar was reduced, but in the last reheat or two the whole length of the bar was worked to the final diameter of that swage. In particular, in the finishing swage, the complete length of the bar was given some work during the final two or three operations.

Finishing temperature of forging was judged by the color of the stock by an experienced forger. An attempt was made to hold this temperature above 1800° F.

Forging characteristics.—During forging an effort was made to evaluate the relative forgeability of the experimental alloys. This evaluation consisted of observing the forging characteristics of the alloys—ease of reduction and tendency to form cracks—and of keeping complete records of the number of reheats and blows required to forge each alloy. These methods were at best only very approximate.

Since the small hammer used was taxed to capacity during the initial stages of the breakdown by the rigidity of most of the alloys and since the force of the blows was varied somewhat, depending on the forgeability of the alloys, the method of comparing the required blows was usable only for distinguishing between alloys with wide differences in plasticity. Judging the relative response of the alloys to the blows from the hammer seemed to be the best method to evaluate forgeability. Relative forgeability could be only qualitatively estimated, however, by this method, because only two ingots were forged together and only a few forged in 1 day.

In table V a summary is given of forging data and the estimated forgeability of all the alloys compared with that of the basic alloy. This forgeability is listed merely as "better," "poorer," or the "same" as that of the basic alloy. In cases where the alloys are either poorer or better, remarks are given to explain this rating.

On the basis of these forgeability ratings the following effects of composition on forgeability were evident:

(1) Additions of Si (0.5 to 1.6 percent), Mo (0 to 7 percent), and N (0.004 to 0.18 percent) had no observable effect on forgeability.

(2) It appeared that Mn (0 to 2.5 percent) increased forgeability by lowering the tendency of cracking.

(3) Additions of C (0.08 to 0.60 percent) decreased forgeability by lowering the plasticity and increasing the tendency of cracking.

(4) The elements Ni (0 to 30 percent), Co (0 to 32 percent), and Cr (10 to 30 percent) lowered plasticity and thus forgeability, Cr seemingly having the greatest effect.

(5) Additions of W (0 to 7 percent) lowered plasticity and forgeability.

(6) The effect of Cb (0 to 6 percent) was unique among the elements. Alloys without Cb were subject to severe cracking during the initial breakdown of the ingot between flat dies, the forgeability thus being poorer than that for the basic alloy. Increasing Cb to the 1 percent of the basic alloy seemed completely to alleviate this situation. When it was increased to 4 and 6 percent the material was more difficult to forge as a result of the decreased plasticity.

HEAT TREATMENT

Originally a solution treatment at 2100° F was considered to be a satisfactory condition for testing. This was on the basis that for one heat of the basic alloy previously studied (reference 1), for which the solution-treatment temperature was varied from 1800° to 2300° F, a treatment at 2100° F gave slightly higher rupture strengths than the other conditions. Further work, however, indicated that there was poor agreement between properties of two heats in this condition and the best agreement between heats resulted from a 2200° F solution treatment followed by a 1400° F aging treatment. The variability in properties between heats was attributed to the relative residual effects of the hot-working which had not been removed by the 2100° F solution treatment. It appeared that the 2200° F treatment tended to minimize, but did not completely remove, the residual hot-working effects without excessively coarsening the grain size. It was therefore selected as the solution temperature for the alloys in this investigation.

Previous investigation of the basic alloy had shown that at temperatures of solution treatment above 2050° F the 100-hour rupture test elongation was quite low and decreased

with increasing solution temperatures. Accompanying this low elongation was a tendency for the alloy to be sensitive to stress concentrations at the specimen fillets, fractures occurring in these regions. However, proper aging of the solution-treated alloy eliminated this stress-concentration sensitivity and increased the 100-hour rupture strength slightly while not appreciably affecting 1000-hour strengths. Aging between 1350° and 1500° F did not produce significant changes in properties. A temperature of 1400° F for 24 hours was selected for aging the experimental alloys in this investigation so that variable effects resulting from stress-concentration sensitivity would be minimized and the materials would be in a more stable condition than when only solution-treated.

It is known that the treatment used does not give the highest strengths at high temperatures for the basic alloy and it is not believed that this treatment necessarily would give the best properties for any of the composition modifications studied. But it is believed that use of this treatment, plus the careful control to hold melting and fabrication conditions as constant as possible, has minimized all variable effects on properties except chemical composition in this investigation.

Heat-treatment procedure.—All of the alloys were solution-treated for 1 hour at 2200° F, water-quenched, aged 24 hours at 1400° F, and air-cooled prior to rupture testing. Solution treatments were made in a gas-fired furnace and aging was in an electric resistance furnace.

Heat-treatment observations.—An observation made during heat treatment of the effect of Cb on scaling characteristics of the alloys appeared to be significant. In alloys containing four or more percent of Cb relatively heavy scaling occurred during the 1-hour solution treatment at 2200° F. This scaling effect was greater on these high-Cb alloys than on the alloy containing 10 percent Cr which was expected to have the poorest corrosion resistance of the alloys studied.

REPRODUCIBILITY OF RUPTURE PROPERTIES AT 1200° F OF BASIC ALLOY

The final objective of the work on techniques for preparing the experimental alloys was the development of a condition in which the rupture properties at 1200° F would be reproducible from heat to heat of a given composition. The interpretation of significant property variations with chemical composition depends on the range of this reproducibility.

The rupture test characteristics at 1200° F for six heats of the basic alloy, the alloy used to evaluate the preparation procedures, are given in table VI, for both square and round bar stock. The range of reproducibility of rupture properties indicated for the round stock forged by the finally adopted swaging procedure was as follows:

100-hour rupture strength, psi	48,000 to 50,000
1000-hour rupture strength, psi	37,000 to 38,000
100-hour rupture elongation, percent	19 to 25

These ranges are used in interpreting the significant influences of composition variables for the materials forged as rounds. Properties of alloys with variable C and Mn, forged early in the program as square stock and tested as such, are compared with the wider property range indicated in table VI for square stock of the basic alloy.

The stress and rupture-time data for the six heats are plotted in figure 5 with curves drawn which indicate the range in strengths for the round stock. Figure 6 presents in a similar manner the data for stress against creep rate obtained from curves of elongation against time for the rupture tests. The narrower property ranges for the round stock compared with those of the square stock are noted.

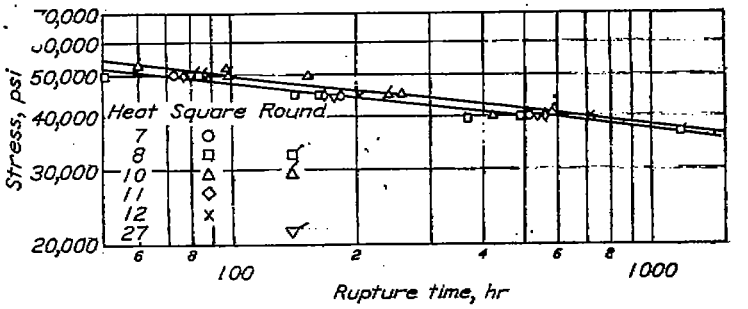


FIGURE 5.—Curves of stress against time for rupture for six heats of basic alloy. Range indicated for round bar stock. Treatment: 2200° F for 1 hour, water-quenched; 1400° F for 24 hours.

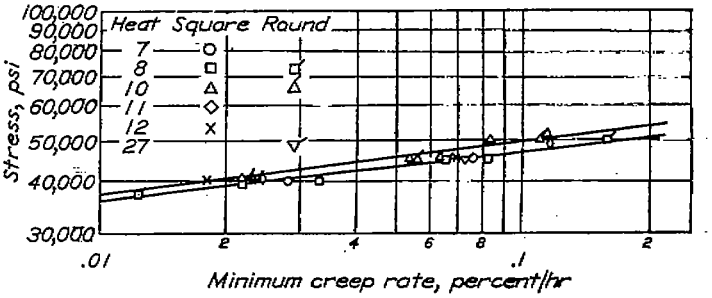


FIGURE 6.—Curves of stress against minimum creep rates at 1200° F for six heats of basic alloy. Range indicated for round bar stock. Treatment: 2200° F for 1 hour, water-quenched; 1400° F for 24 hours.

As an example of the narrowing of the range by using the more easily controlled and duplicated swaging procedure, alloy 8 had the lowest (46,500 psi) and alloy 10 the highest (52,000 psi) 100-hour rupture strengths of the 5500-psi over-all range for square bar stock. After forging as rounds these same two heats had 100-hour rupture strengths of 48,500 and 50,000 psi, thus narrowing the over-all range of 100-hour rupture strengths to 1500 psi. The 1000-hour rupture-strength range was narrowed from 2500 to 1000 psi by changing the forging procedure. Similar changes in ranges for squares and rounds are indicated in table VI for stresses at constant creep rates and for the minimum creep rate at constant stress.

Table VI also gives properties of alloys 74 and 75 which indicate that variation in melting deoxidation practice can have a pronounced effect on high-temperature characteristics of alloys of this type.

The influence on rupture test characteristics of changing hot-working and deoxidation procedures indicates that the solution treatment at 2200° F did not completely eliminate variables in prior processing. However, the use of the same deoxidation practice for all the alloys and the control of hot-working resulting from the adoption of the procedure producing rounds rather than squares plus the use of the 2200° F solution treatment are believed to have minimized the effects of variables in processing on the high-temperature properties.

Data for two commercial heats of the basic alloy with the same heat treatment as the experimental alloys have been included in table VI to show the relationship between the experimental alloys and normal commercial properties.

INFLUENCE OF CHEMICAL-COMPOSITION VARIABLES ON ROOM-TEMPERATURE METALLURGICAL CHARACTERISTICS OF MODIFIED ALLOYS

Microstructural studies and hardness tests were made on all the experimental alloys to obtain metallurgical information to aid in interpretation of the influence of chemical composition on rupture properties.

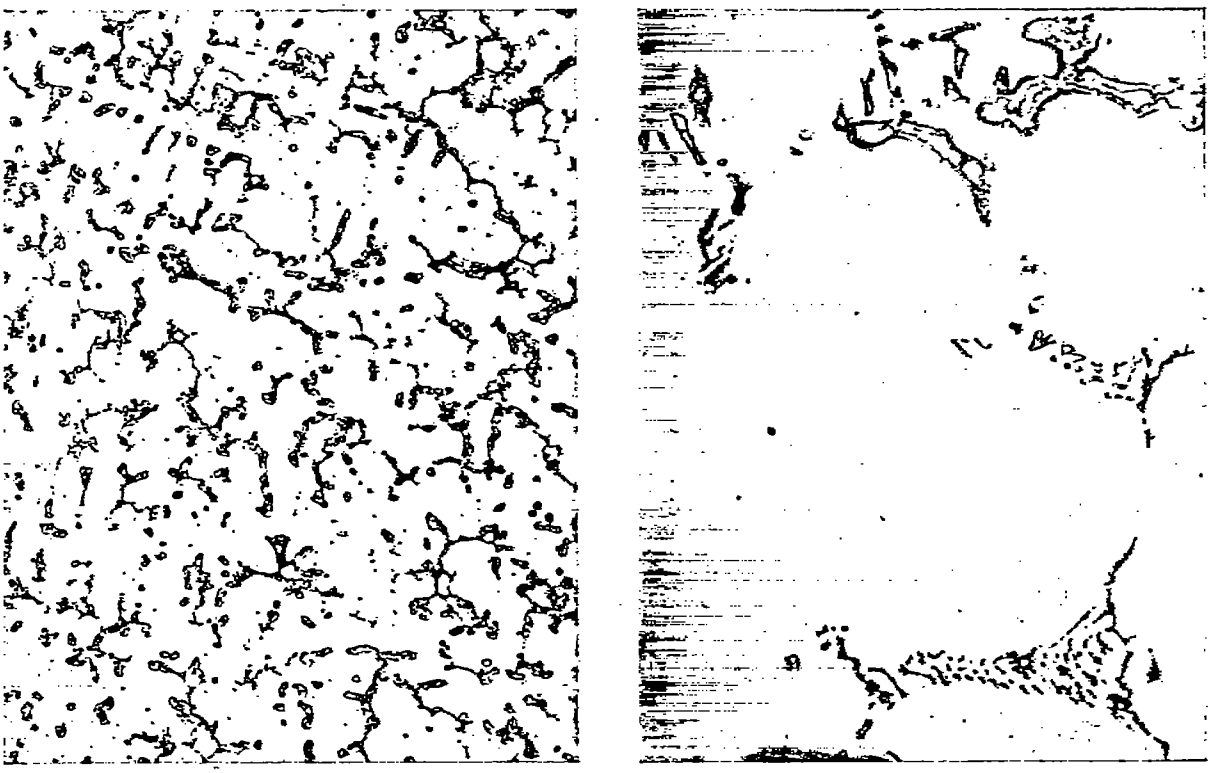
MICROSTRUCTURE

Microstructural studies were made of each of the alloys in the following conditions: As-cast; hot-forged; solution-treated 1 hour at 2200° F and water-quenched; aged 24 hours at 1400° F after solution treatment; and rupture-tested the maximum time at 1200° F. An electrolytic solution of 10 percent chromic acid was used as an etchant.

Effect of treatment on microstructures.—The as-cast structure revealed the amount and distribution of the excess constituents in the cast dendritic pattern. The hot-forged structures were examined for flaws, uniformity of grain size, and distribution of excess constituents. Only limited information is included on the cast and forged structures, the main emphasis being placed on structures of the alloys prior to testing in the heat-treated condition. The solution-treated structure showed the amount of insoluble excess constituents after light etching and the grain size after deep etching. The grain size was much easier to determine in the aged structures because the grain boundaries were revealed by light etching. The aged structure also revealed the amount and mode of matrix precipitation. From the structure of the maximum-time rupture test any change occurring during testing and the mode of fracture could be observed.

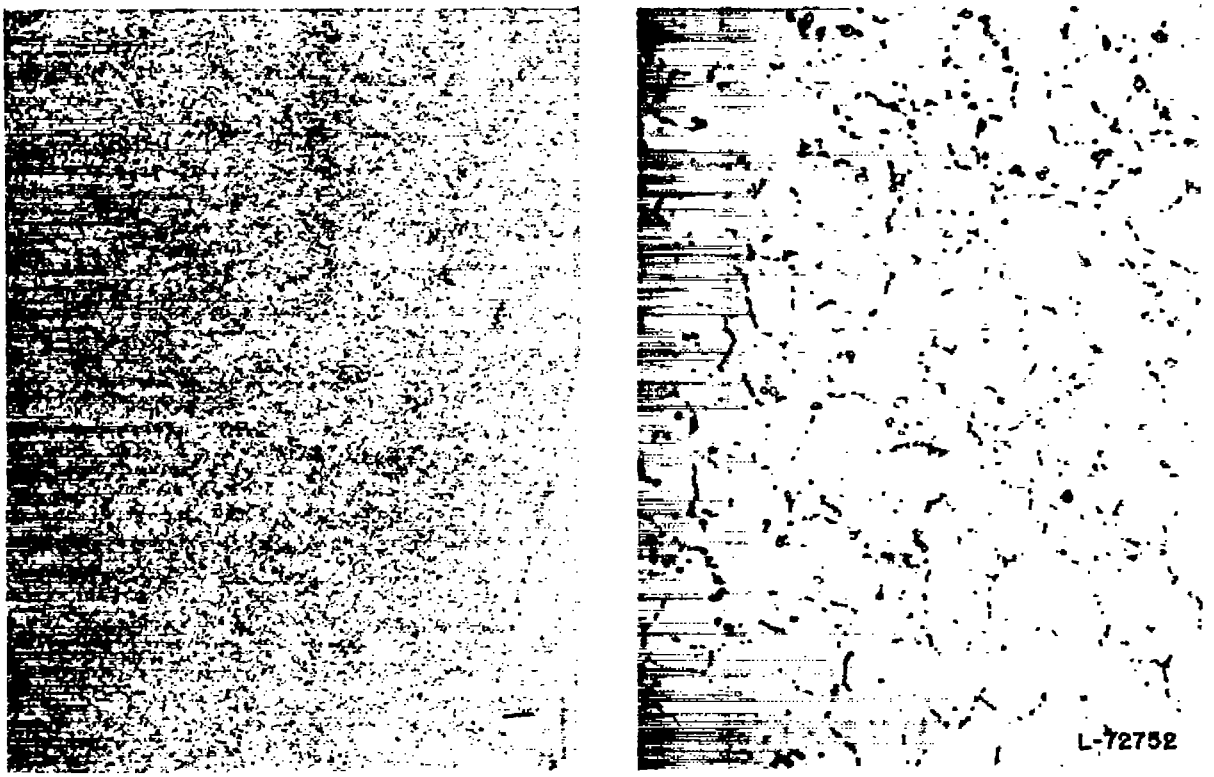
An example of microstructural variation with treatment for the basic alloy is shown in figure 7. The as-cast condition contained a considerable amount of excess constituent. After hot-forging the alloy was uniformly fine-grained and contained excess constituent at the grain boundaries and in the matrix. Solution-treating at 2200° F dissolved the grain boundary constituent but not the matrix constituent. Solution-treating also coarsened the grains considerably as is best shown by the aged structure. Aging caused considerable grain boundary precipitate and some random matrix precipitation. Rupture testing at 1200° F increased the matrix precipitate slightly.

As an example of one extreme to which microstructures varied with composition, figure 8 shows microstructures for the 0Mo–0W–0Cb alloy in the as-cast, solution-treated, aged, and rupture-tested conditions. The structures differed from those of the basic alloy in that: There was much less excess constituent in the as-cast and the solution-treated conditions; grain size after solution treatment was much larger; and aging produced more precipitate near the grain boundaries and a preferred type of matrix precipitate which tended to follow definite crystallographic planes. The two alloys also differed in that the 0Mo–0W–0Cb alloy had a completely intergranular fracture while that of the basic alloy was approximately half intergranular and half transgranular.



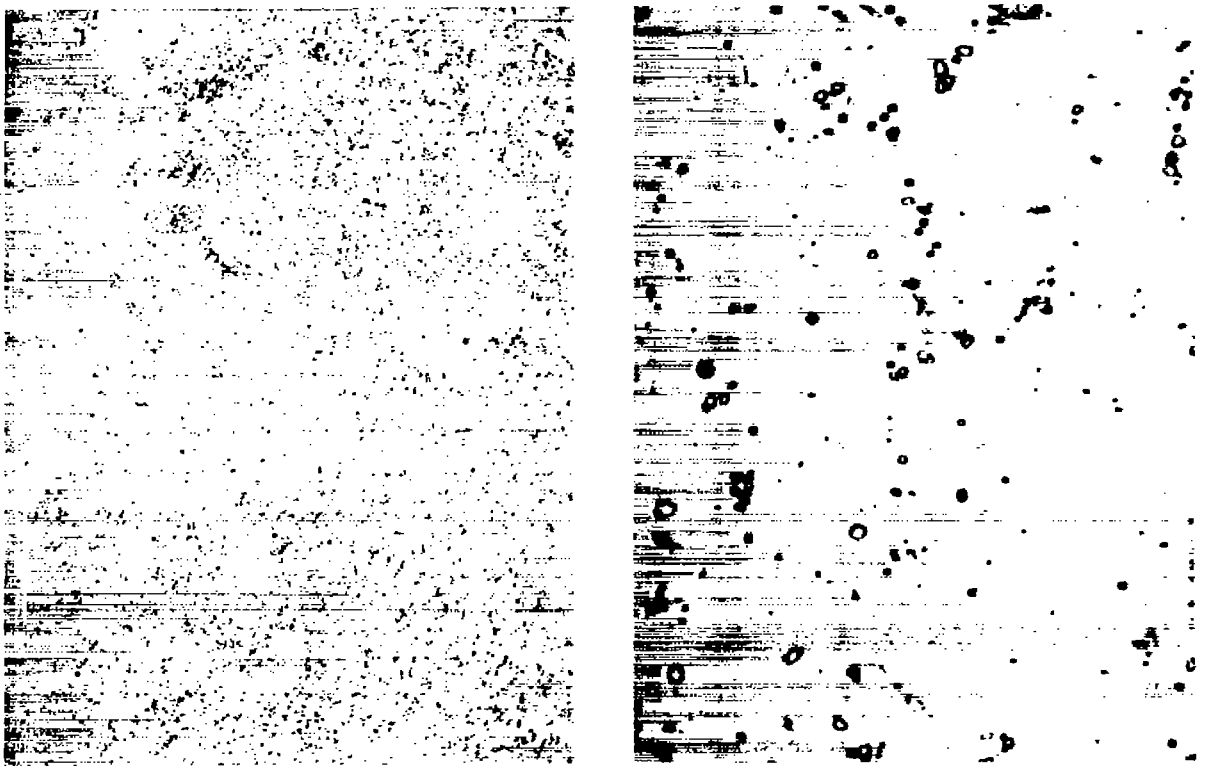
100X (a) Ingot. 1000X

FIGURE 7.—Microstructures of basic alloy (heat 8).



100X 1000X

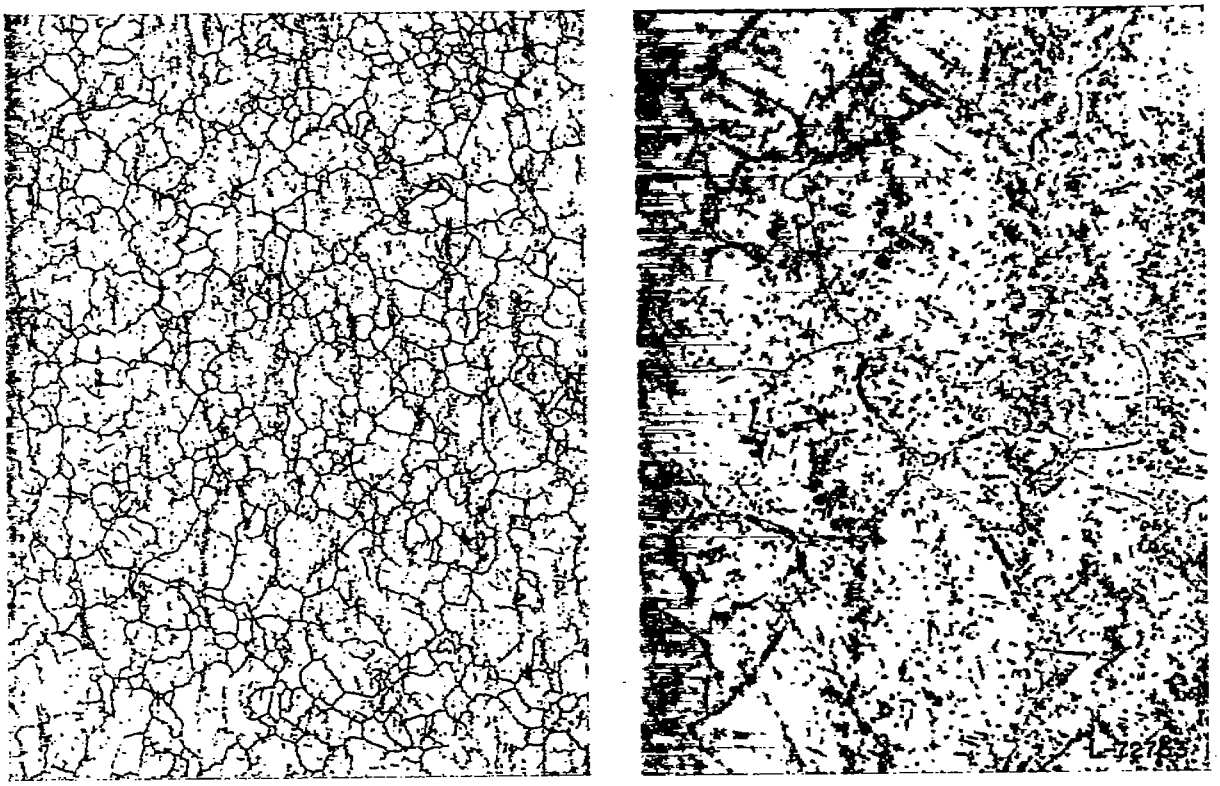
(b) As-forged 0.4-inch-diameter bar stock.



100X 1000X

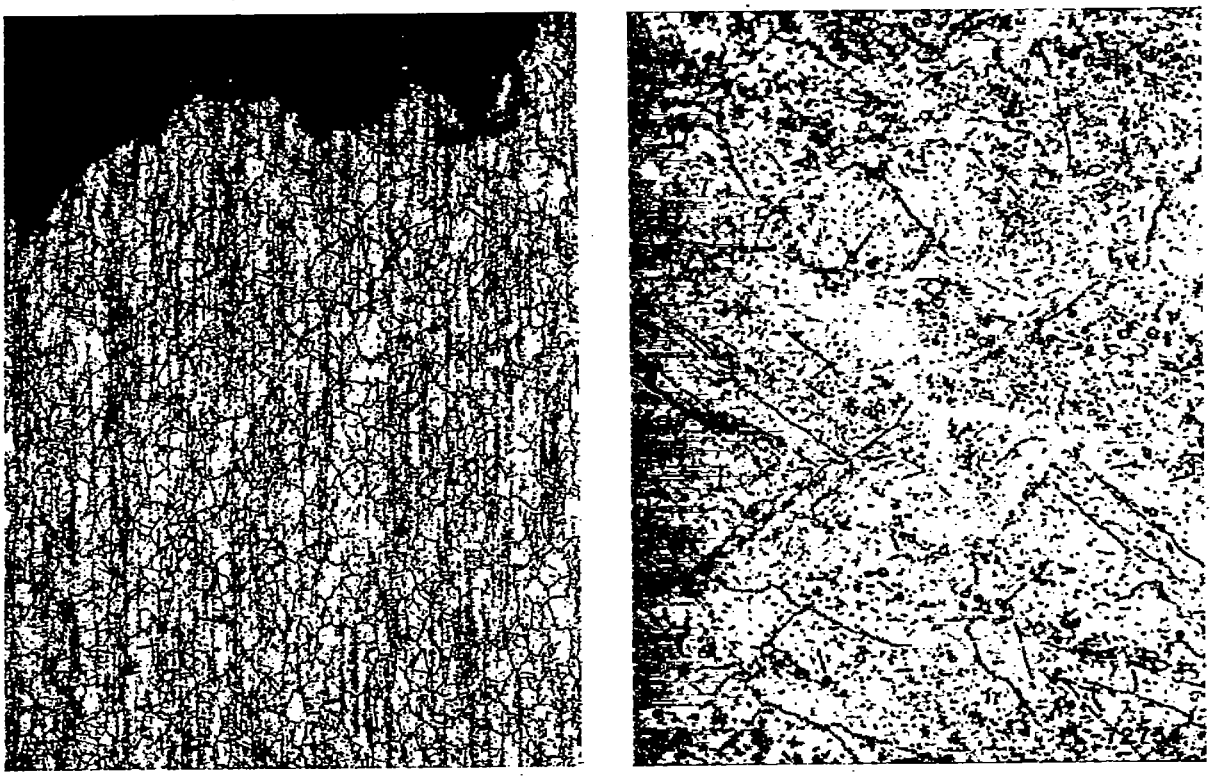
(c) Solution-treated; 1 hour at 2200° F, water-quenched.

FIGURE 7.—Continued.



100X 1000X

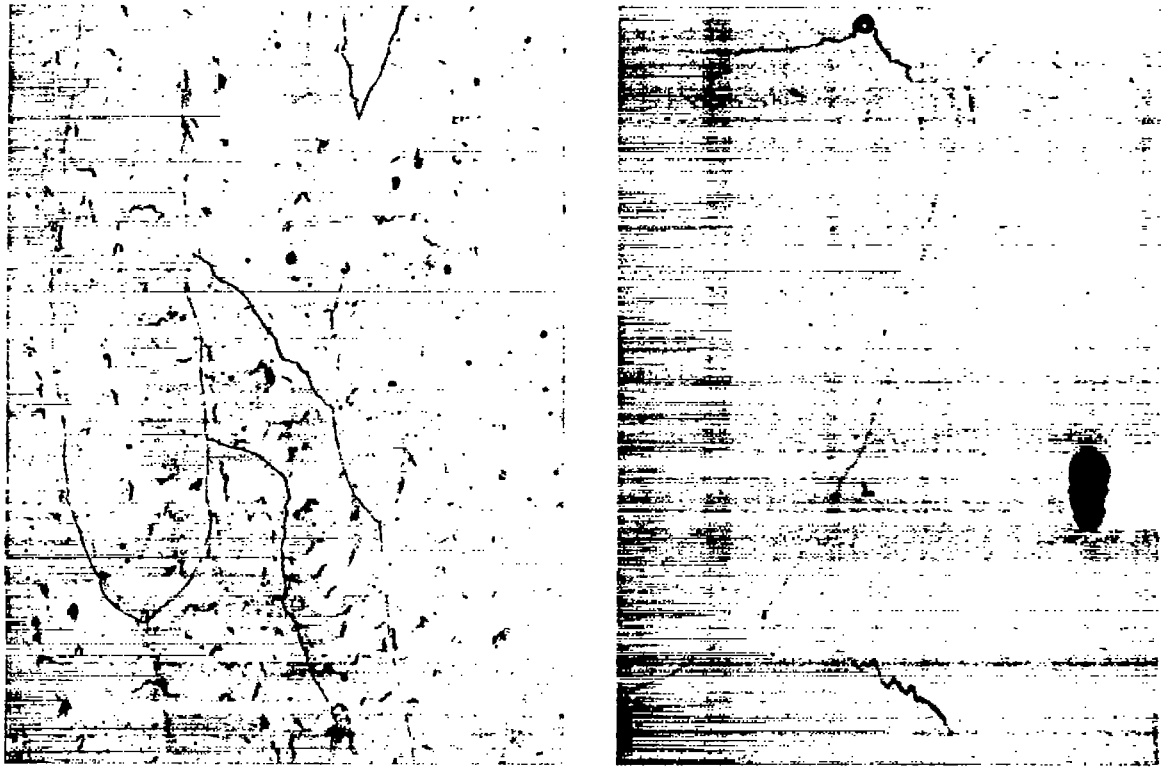
(d) Solution-treated and aged 24 hours at 1400° F.



Fracture 100X 1000X

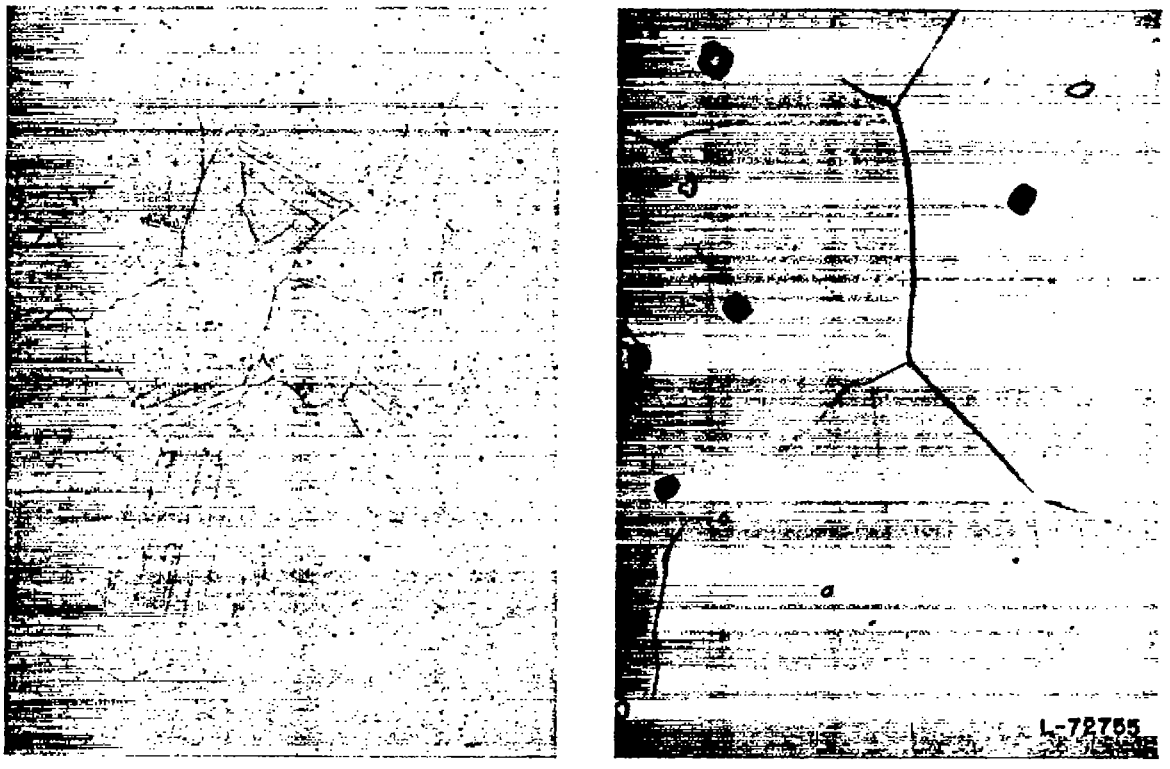
(e) Rupture specimen; 499 hours for rupture at 1200° F under 40,000 psi.

FIGURE 7.—Concluded.



100X 1000X

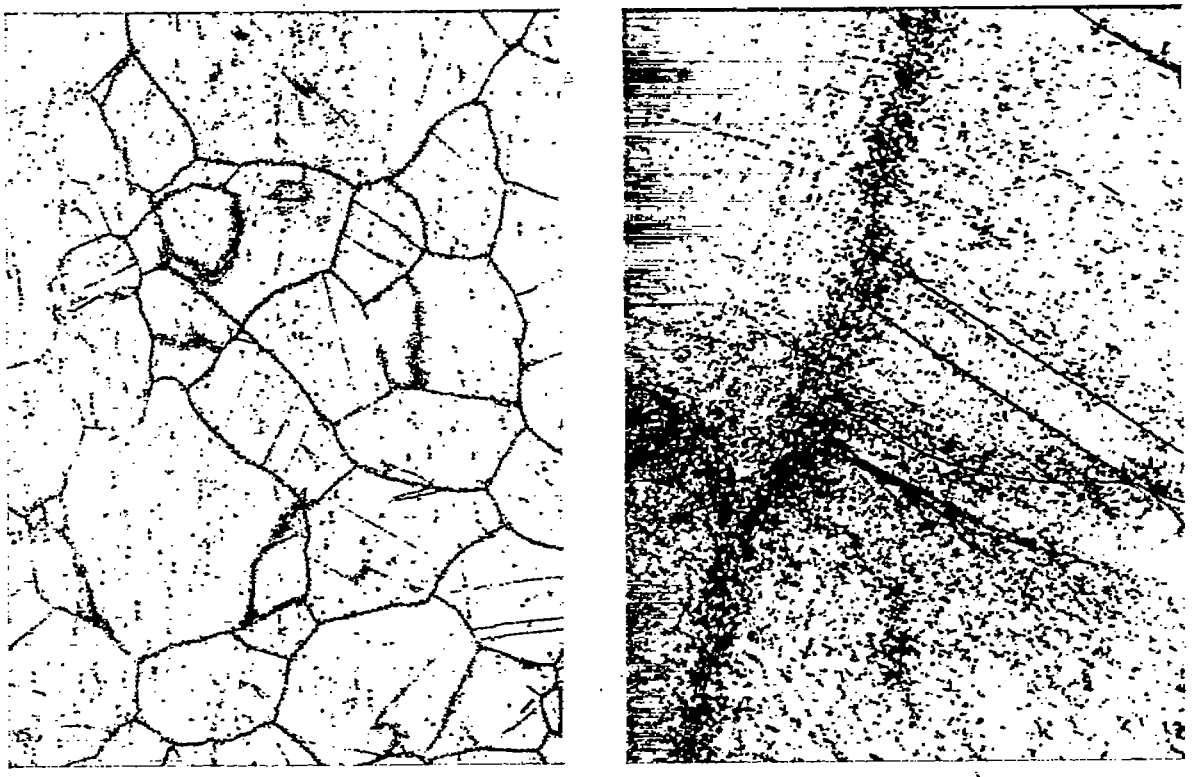
(a) Ingot.



100X 1000X

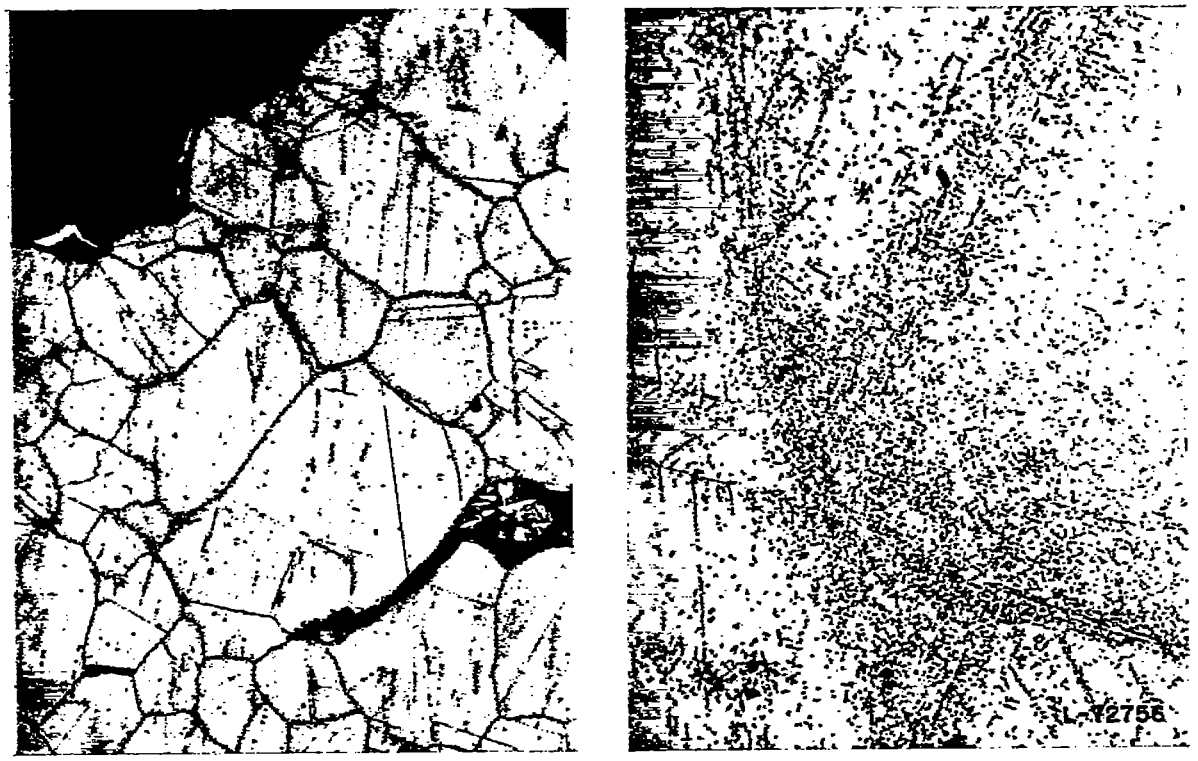
(b) Solution-treated; 1 hour at 2200° F, water-quenched.

FIGURE 8.—Effect on microstructure of omitting molybdenum, tungsten, and columbium from basic analysis (heat 43).



100X 1000X

(c) Solution-treated and aged 24 hours at 1400° F.



100X 1000X

(d) Rupture specimen; 819 hours for rupture at 1200° F under 20,000 psi.

FIGURE 8.—Concluded.

The above comparison has pointed out the major similarities and differences in structures which were found for all the alloys. Similarities were:

(1) The amount of excess constituent in the solution-treated condition was proportionate, for any given alloy, to the amount of excess constituent in the as-cast condition. The structures indicate that the rapid cooling of the ingot after solidification effectively solution-treated the material.

(2) All grain boundary constituents were dissolved during solution treatment.

(3) Precipitation occurred during aging at 1400° F.

(4) Very little additional precipitation occurred during rupture testing at 1200° F for any of the alloys with the exception of low-Co modifications.

Major structural differences between the alloys were:

(1) Amount of excess constituent after solution treatment.

(2) Grain size.

(3) Location, type, and amount of aging precipitate.

Classification of microstructures.—In order to condense the description of and to make possible a quick comparison of the changes in microstructure occurring with variation in chemical composition the following classification of microstructures of the solution-treated and the aged materials was devised. Symbols were used to indicate the differences in the basic structural characteristics.

(1) Solution-treated structures

Amount of insoluble constituent:

I—small
II—medium
III—large
IV—very large

(2) Aged structures

A. S. T. M. grain size number:

1 (up to 1½ grains/sq in. at 100X magnification) to 8 (96 grains or more/sq in. at 100X magnification)

Amount of grain boundary precipitate:

x—small
y—medium
z—large

Type of matrix precipitate:

C—precipitate tends to follow crystallographic planes
R—random matrix precipitate

Amount of matrix precipitate:

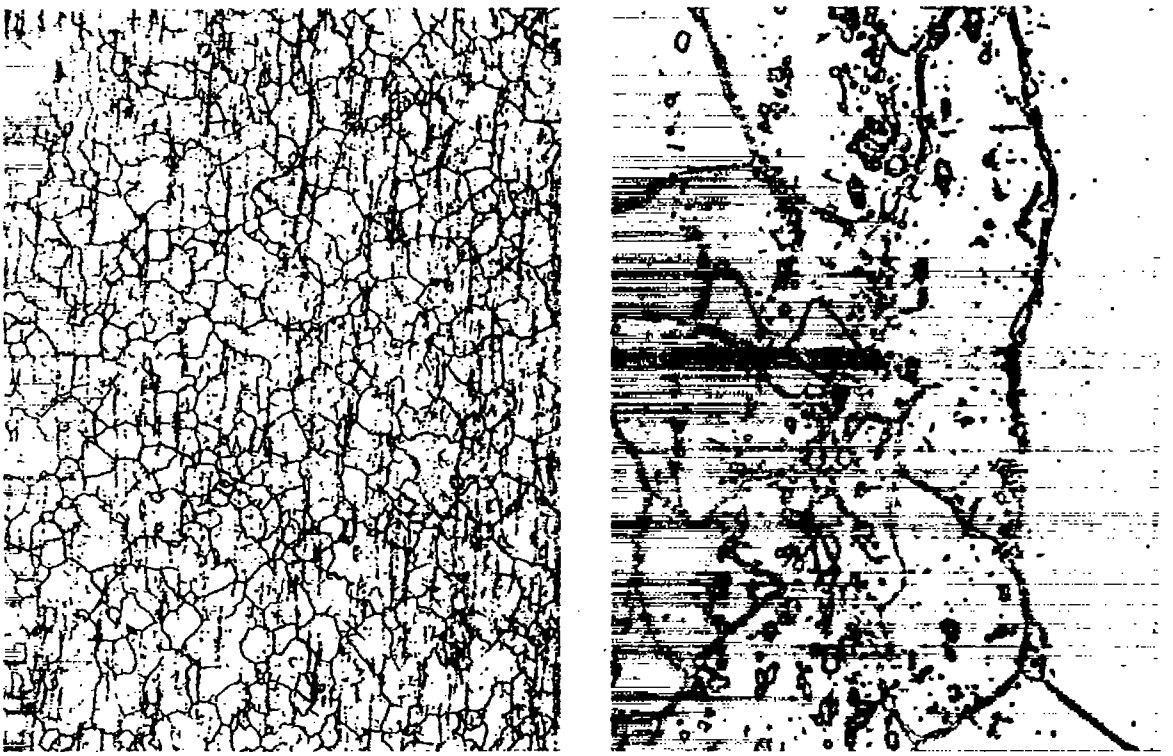
a—small
b—medium
c—large
d—very large

As an example of how this classification works the structures of the basic alloy (fig. 7) and the 0Mo-0W-0Cb modification (fig. 8) are classified below:

(1) Basic alloy:

Classification—II 5 y Rb

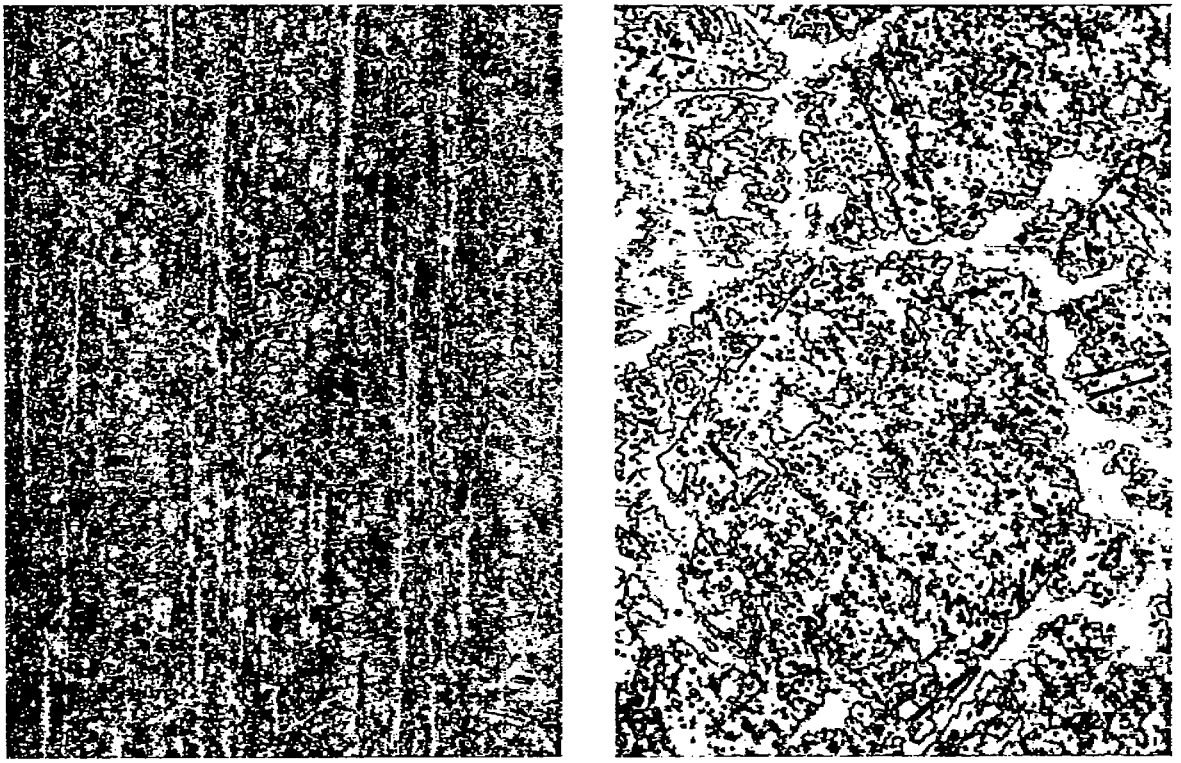
II—medium amount of insoluble constituent
5—number 5 A. S. T. M. average grain size
y—medium amount of precipitate near the grain boundaries
Rb—medium (b) amount of random (R) matrix precipitate



100X 1000X

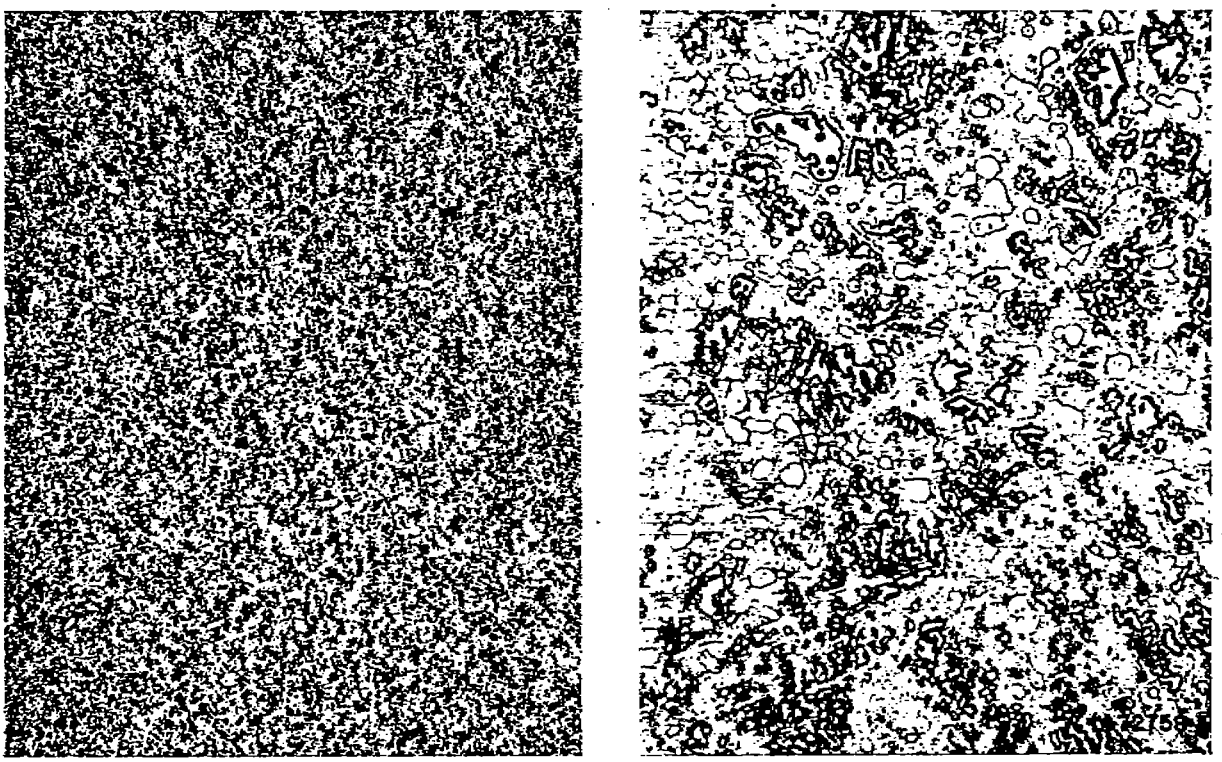
(a) Carbon, 0.08 percent (heat 13).

FIGURE 9.—Influence of carbon content on microstructure of solution-treated and aged basic alloy. Treatment: 2200° F for 1 hour, water-quenched; 1400° F for 24 hours.



100X 1000X

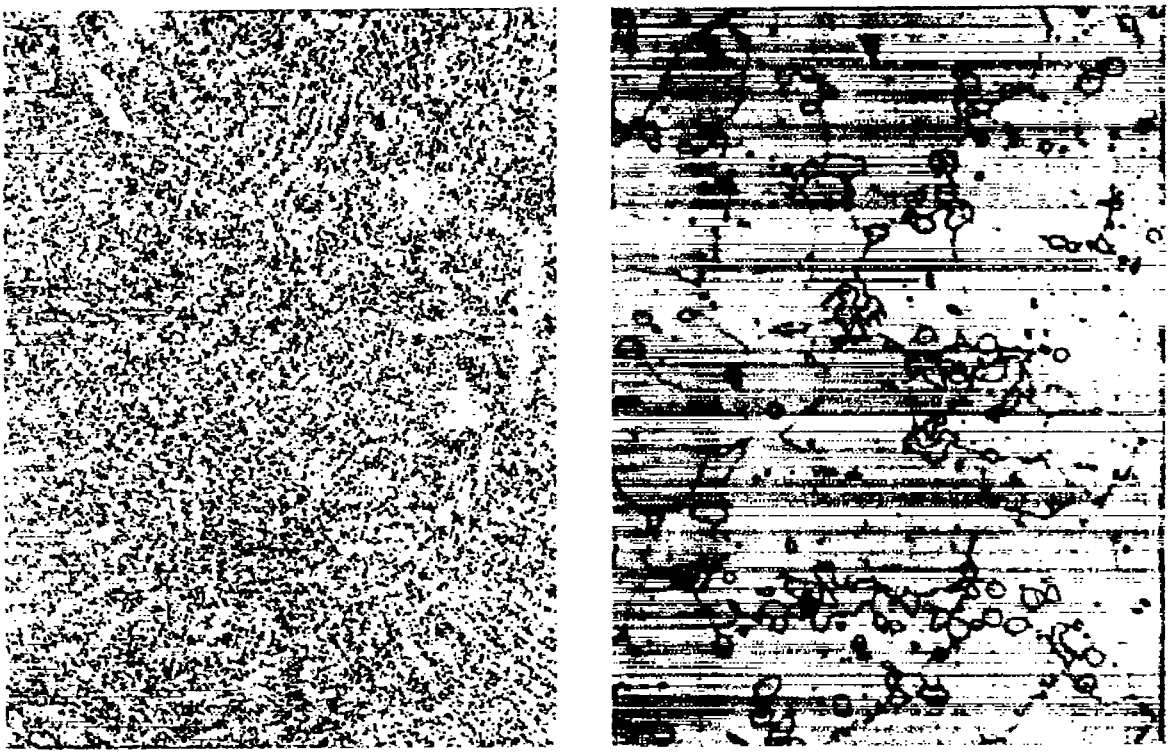
(b) Carbon, 0.40 percent (heat 15).



100X 1000X

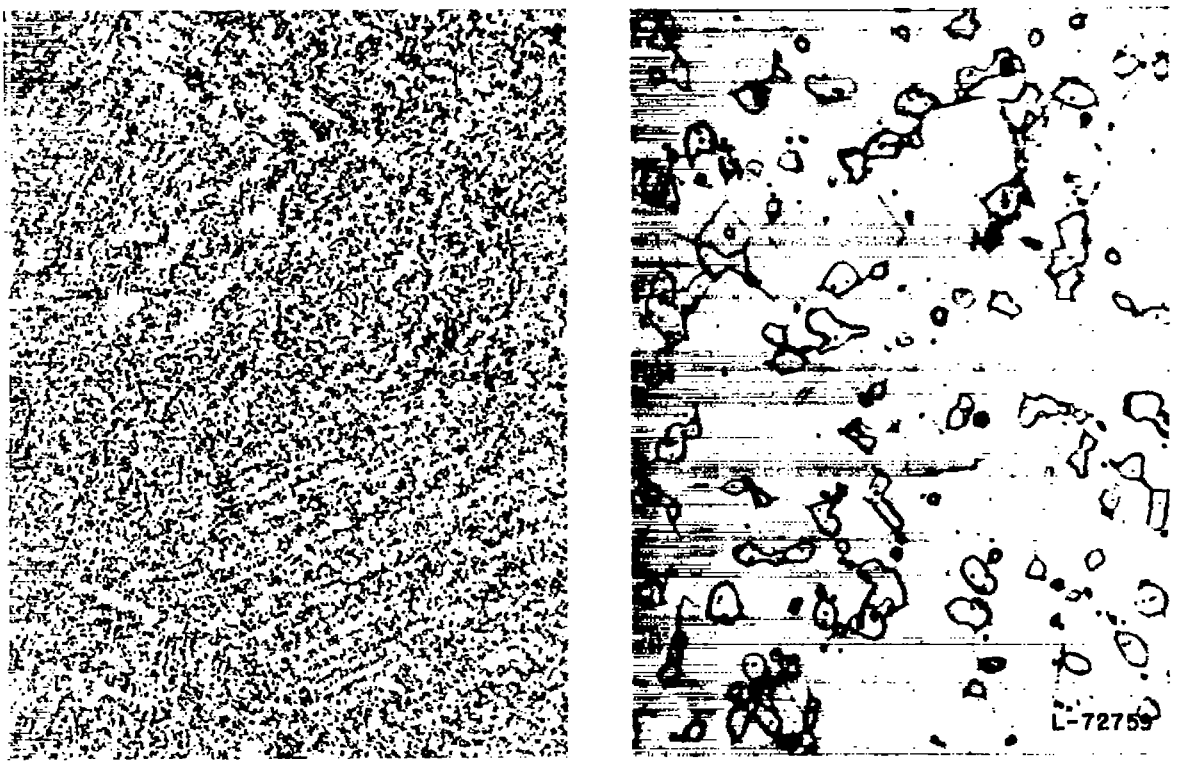
(c) Carbon, 0.60 percent (heat 16).

FIGURE 9.—Concluded.



100X 1000X

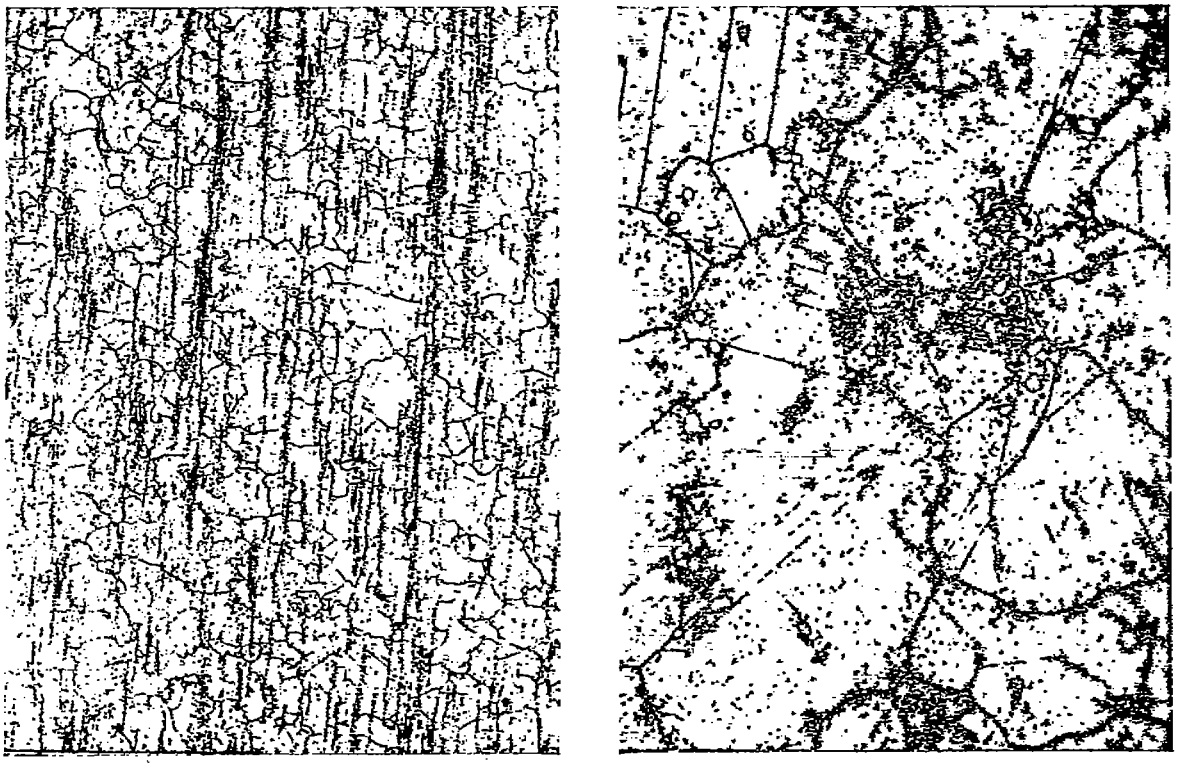
(a) 1 hour at 2200° F, water-quenched.



100X 1000X

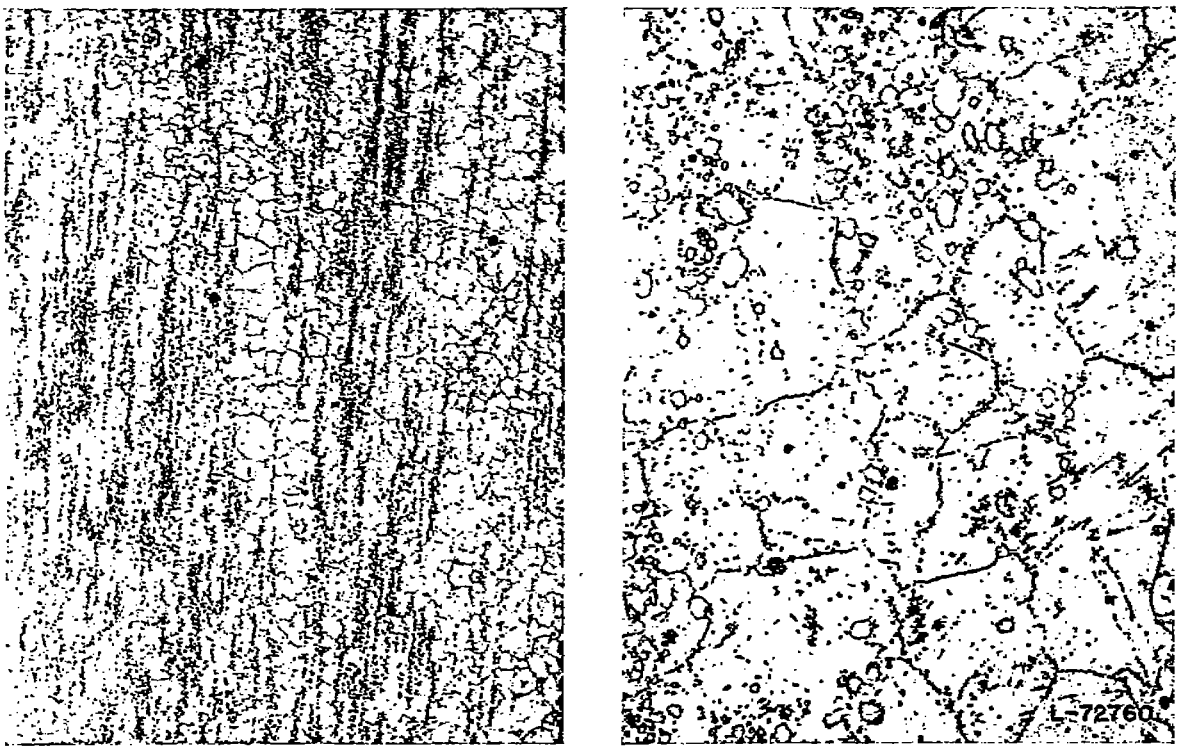
(b) 10 hours at 2200° F, water-quenched.

FIGURE 10.—Effect of increasing solution time on microstructure of 0.60-percent-carbon basic alloy (heat 16).



100X 1000X

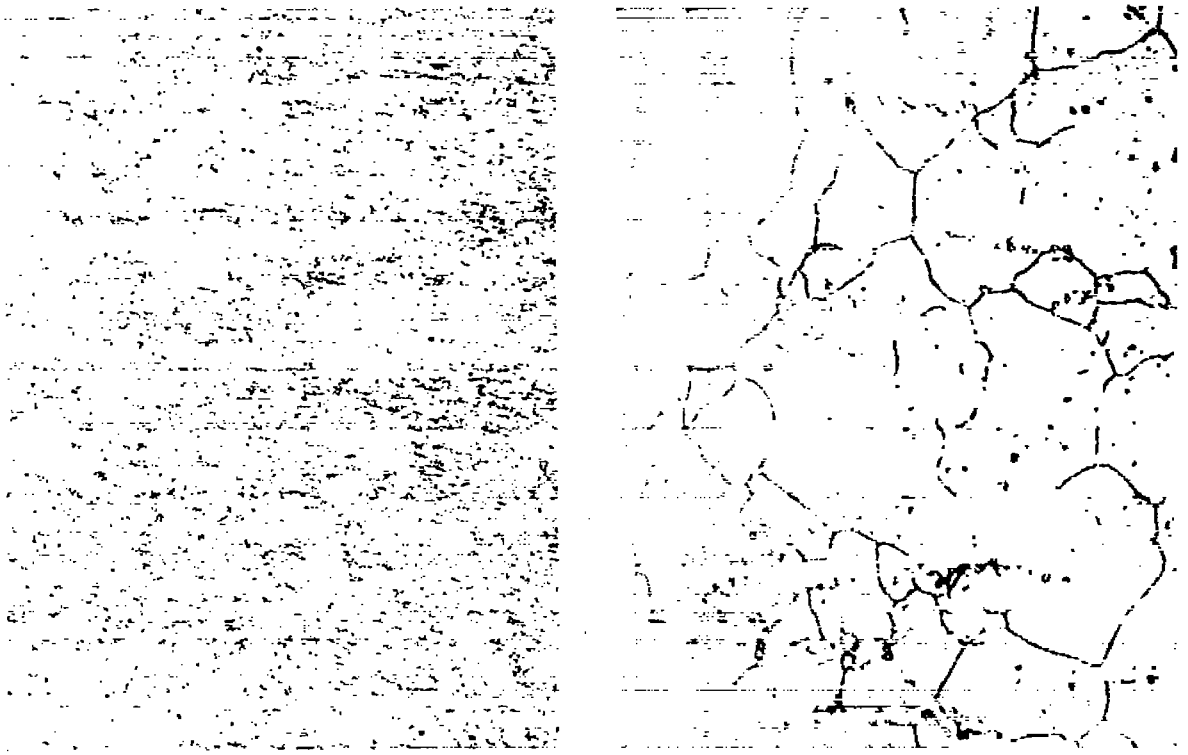
(a) Silicon, 1.2 percent (heat 28).



100X 1000X

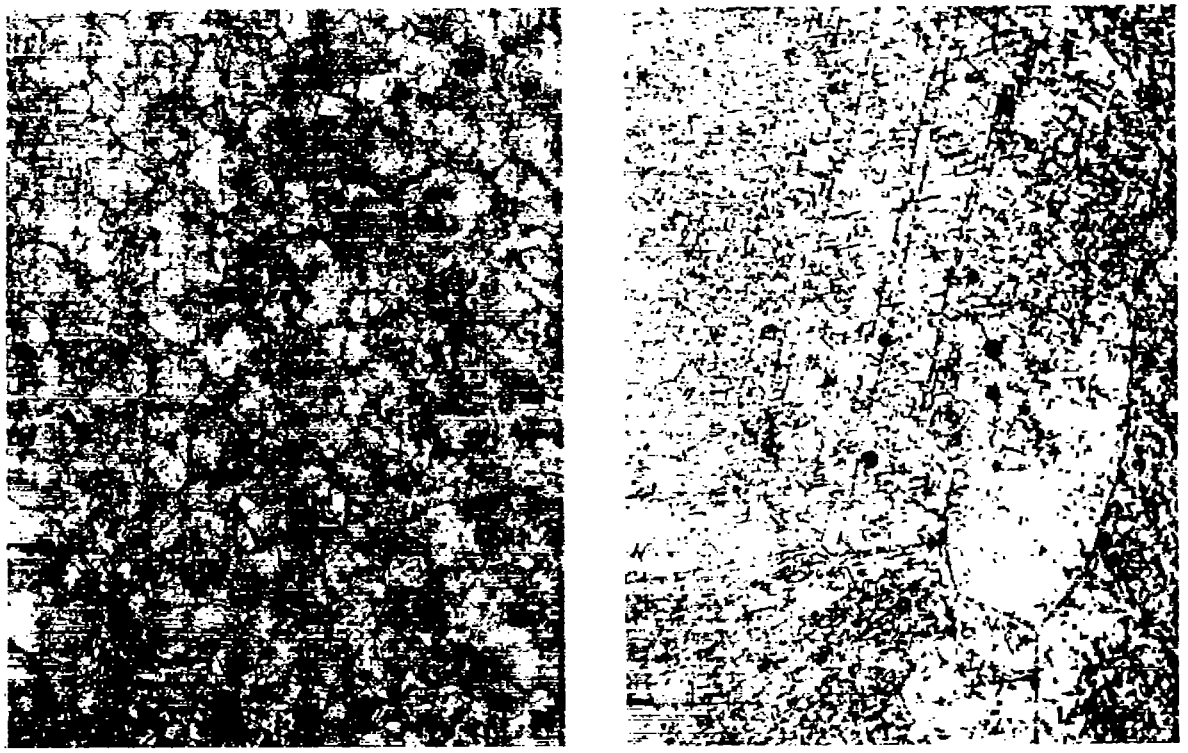
(b) Silicon, 1.6 percent (heat 80).

FIGURE 11.—Influence of silicon content on microstructure of solution-treated and aged basic alloy. Treatment: 2200° F for 1 hour, water-quenched; 1400° F for 24 hours.



100X 1000X

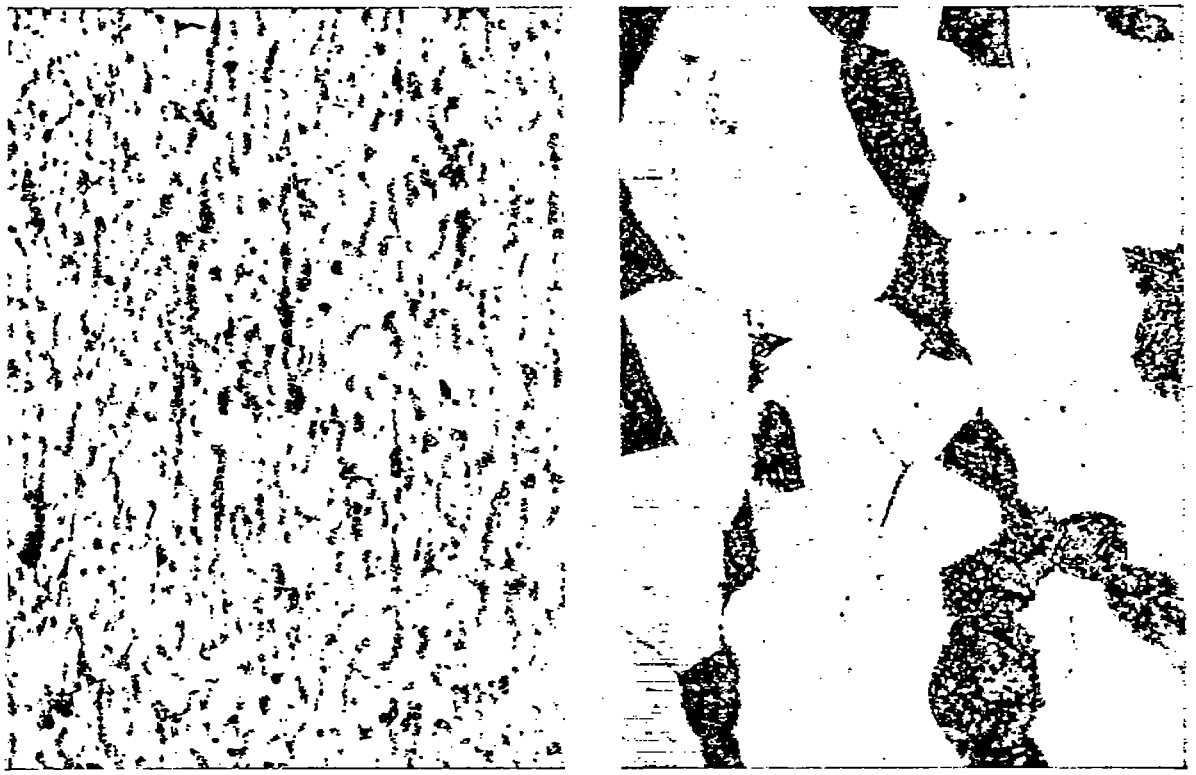
(a) Chromium, 10 percent (heat 51).



100X 1000X

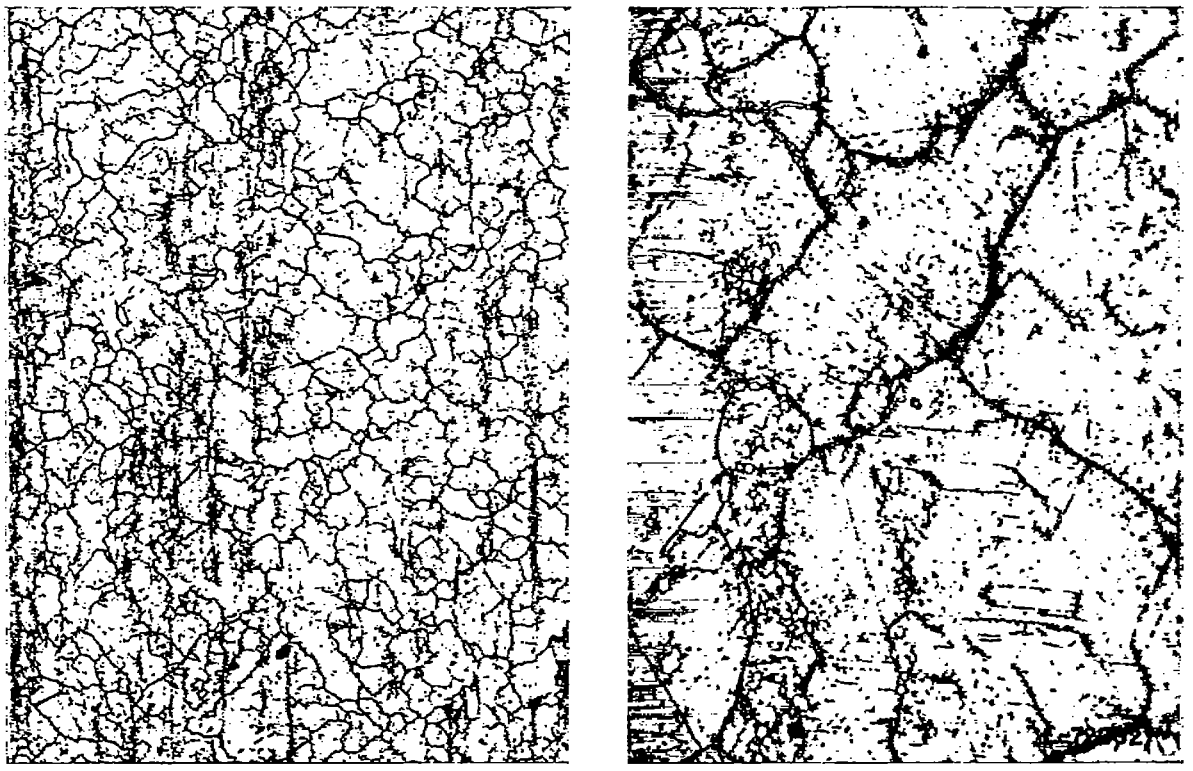
(b) Chromium, 30 percent (heat 52).

FIGURE 12.—Influence of chromium content on microstructure of solution-treated and aged basic alloy. Treatment: 2200° F for 1 hour, water-quenched; 1400° F for 24 hours.



100X 1000X

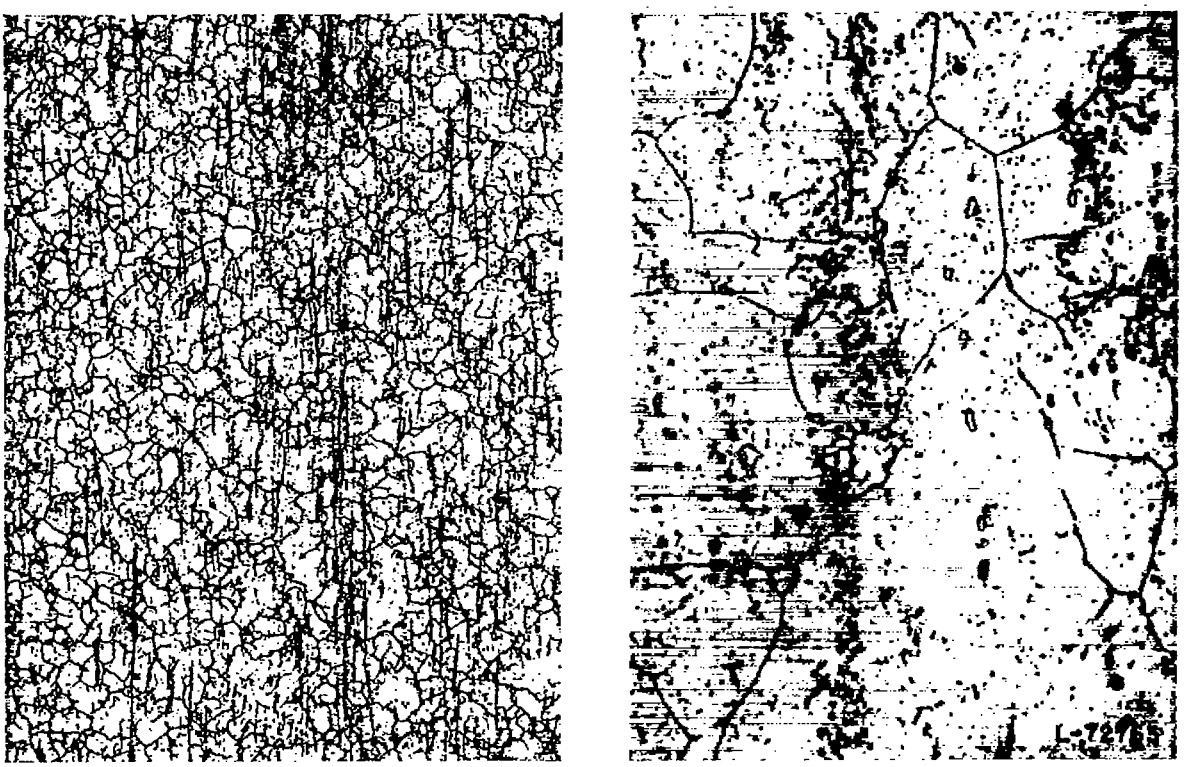
(a) Nickel, 0 percent (heat 23).



100X 1000X

(b) Nickel, 10 percent (heat 25).

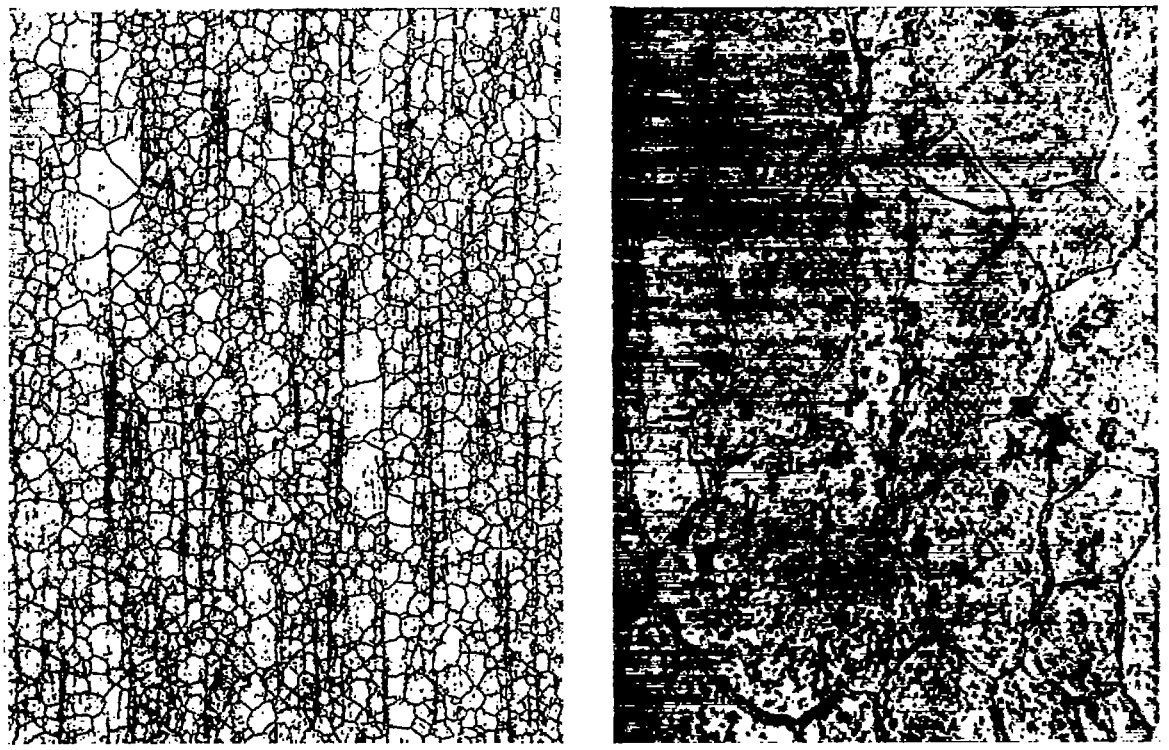
FIGURE 13.—Influence of nickel content on microstructure of solution-treated and aged basic alloy. Treatment: 2200° F for 1 hour, water-quenched; 1400° F for 24 hours.



100X 1000X

(c) Nickel, 30 percent (heat 26).

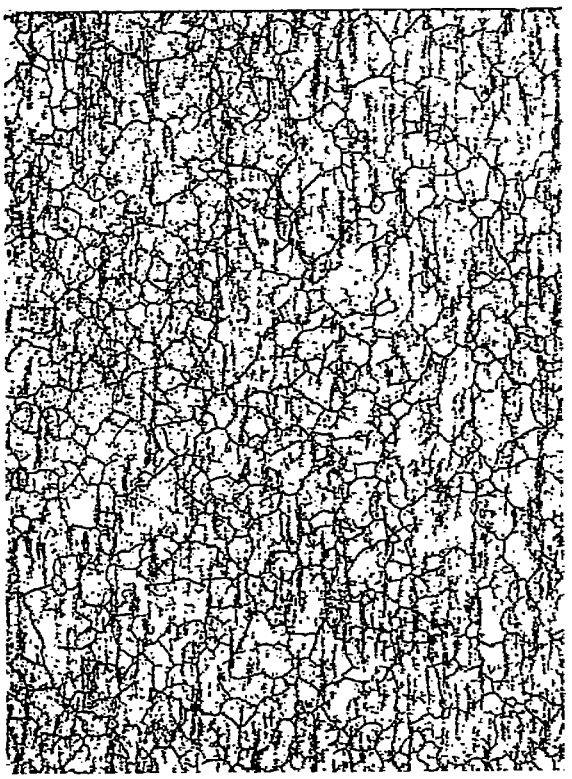
FIGURE 13.—Concluded.



100X 1000X

(a) Cobalt, 0 percent (heat 29).

FIGURE 14.—Influence of cobalt content on microstructure of solution-treated and aged basic alloy. Treatment: 2200° F for 1 hour, water-quenched; 1400° F for 24 hours

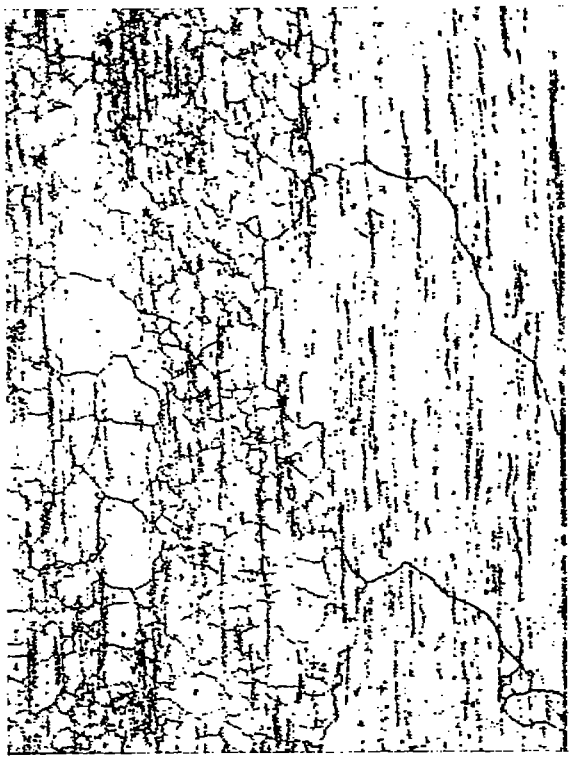


100X



1000X

(b) Cobalt, 10 percent (heat 30).



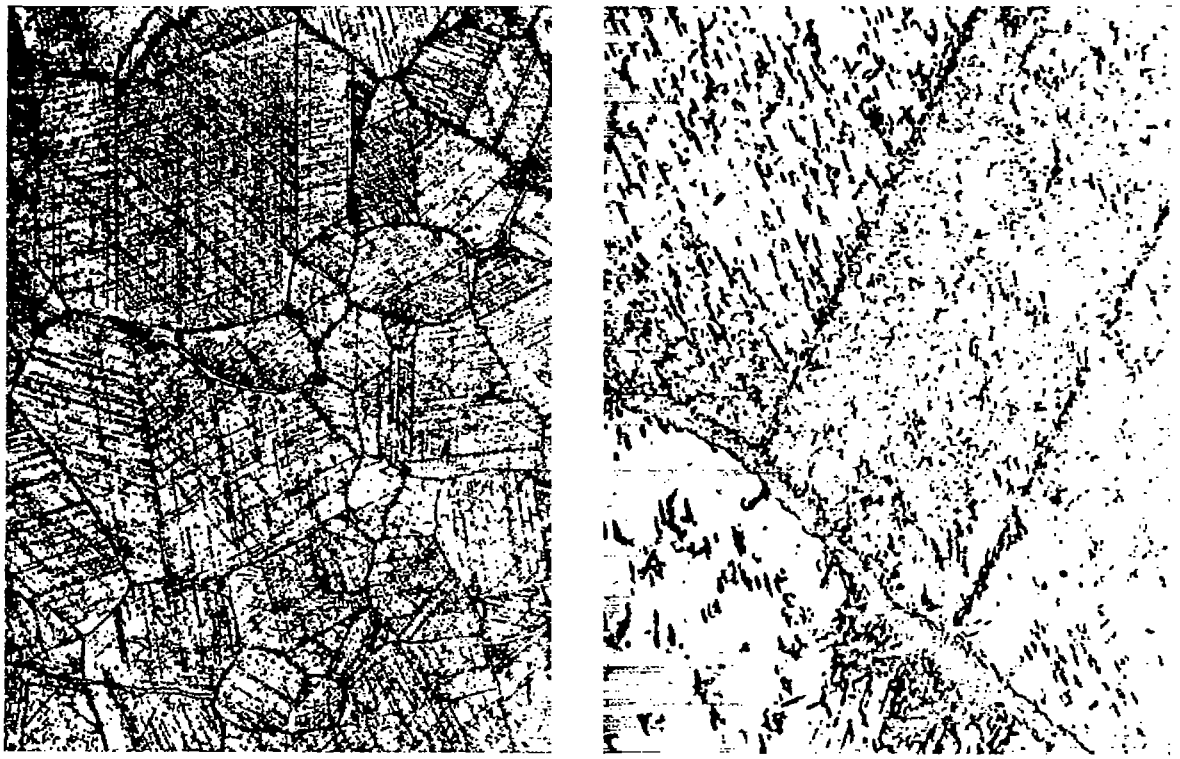
100X



1000X

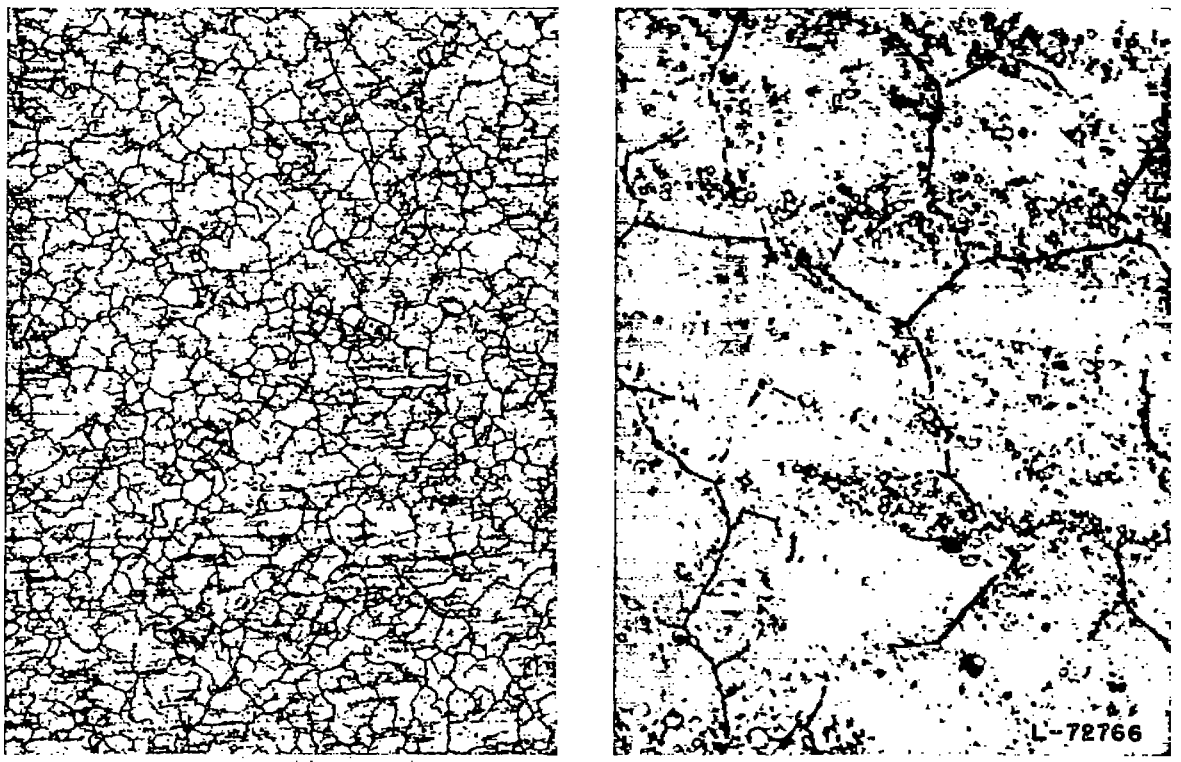
(c) Cobalt, 32 percent (heat 31).

FIGURE 14.—Concluded.



100X 1000X

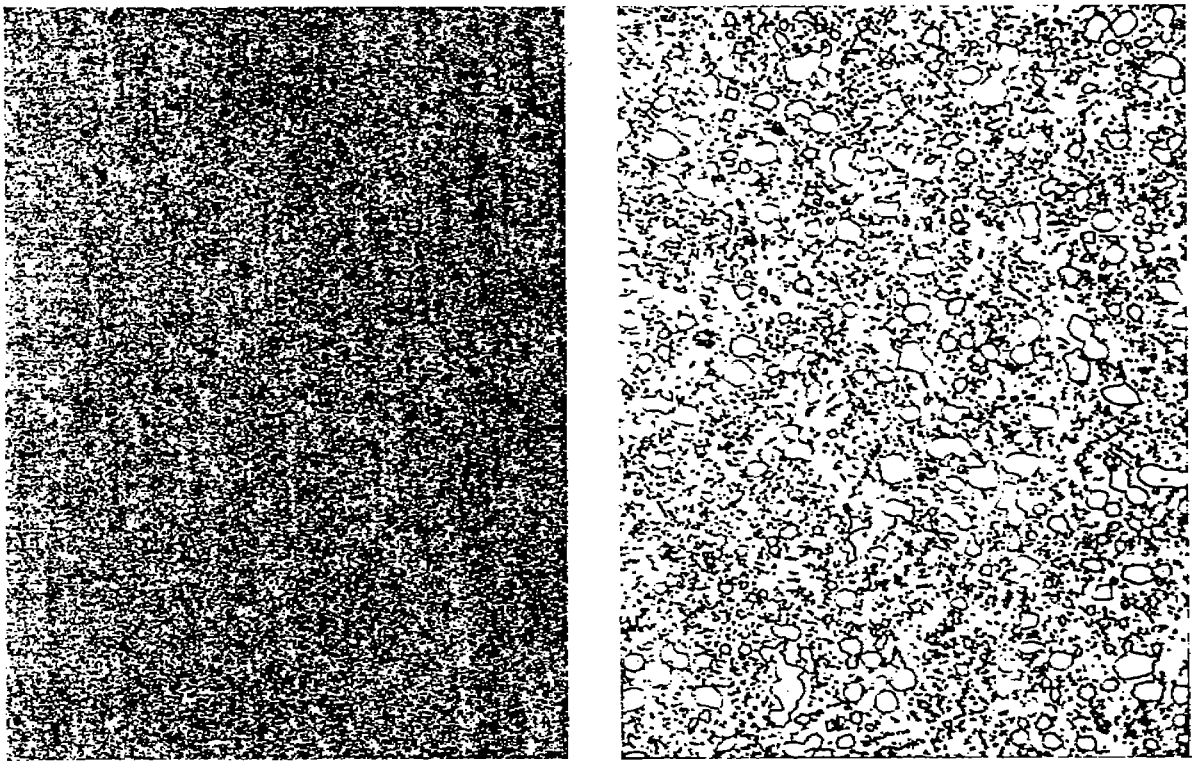
(a) Columbium, 0 percent (heat 47).



100X 1000X

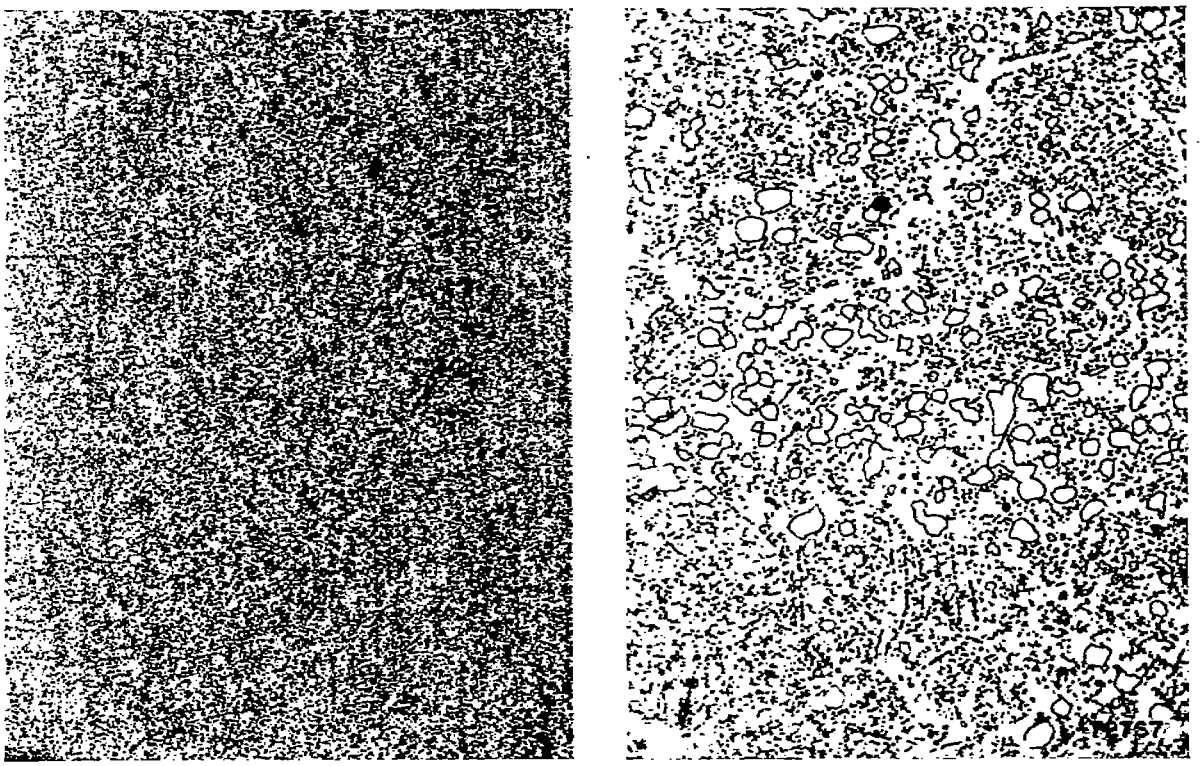
(b) Columbium, 2 percent (heat 42).

FIGURE 15.—Influence of columbium content on microstructure of solution-treated and aged basic alloy. Treatment: 2200° F for 1 hour, water-quenched; 1400° F for 24 hours.



100X 1000X

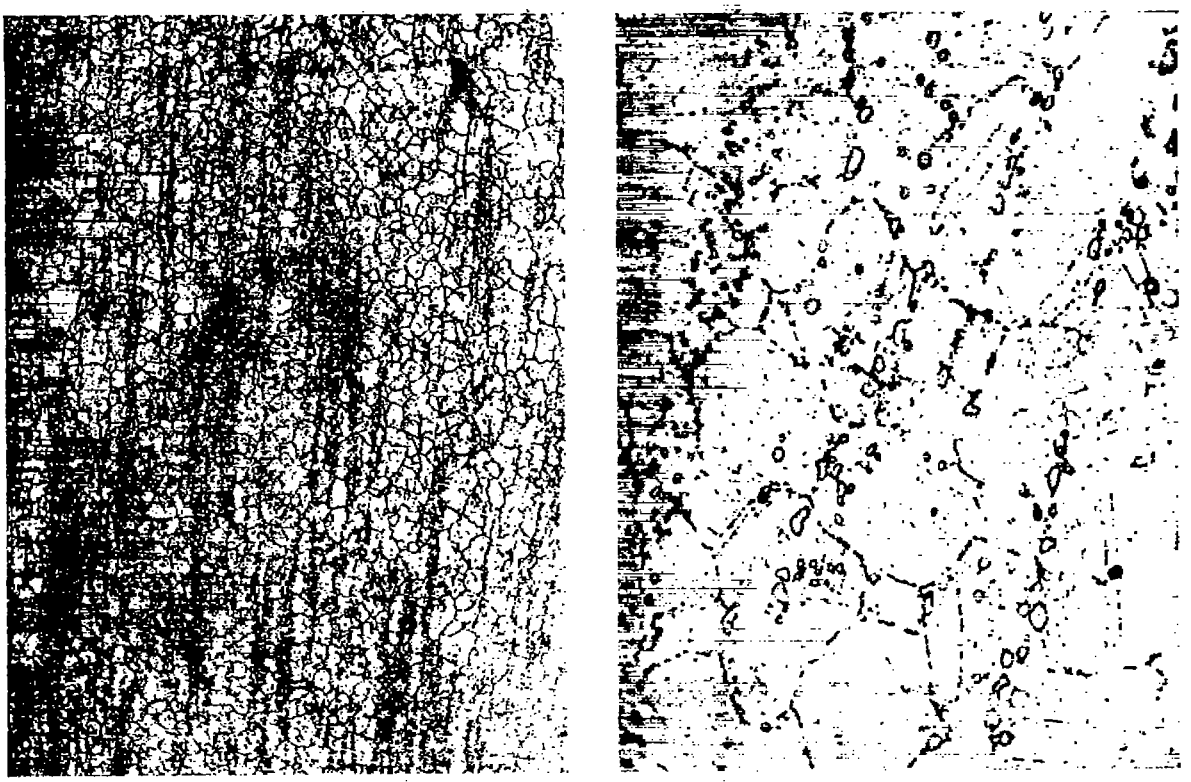
(c) Columbium, 4 percent (heat 49).



100X 1000X

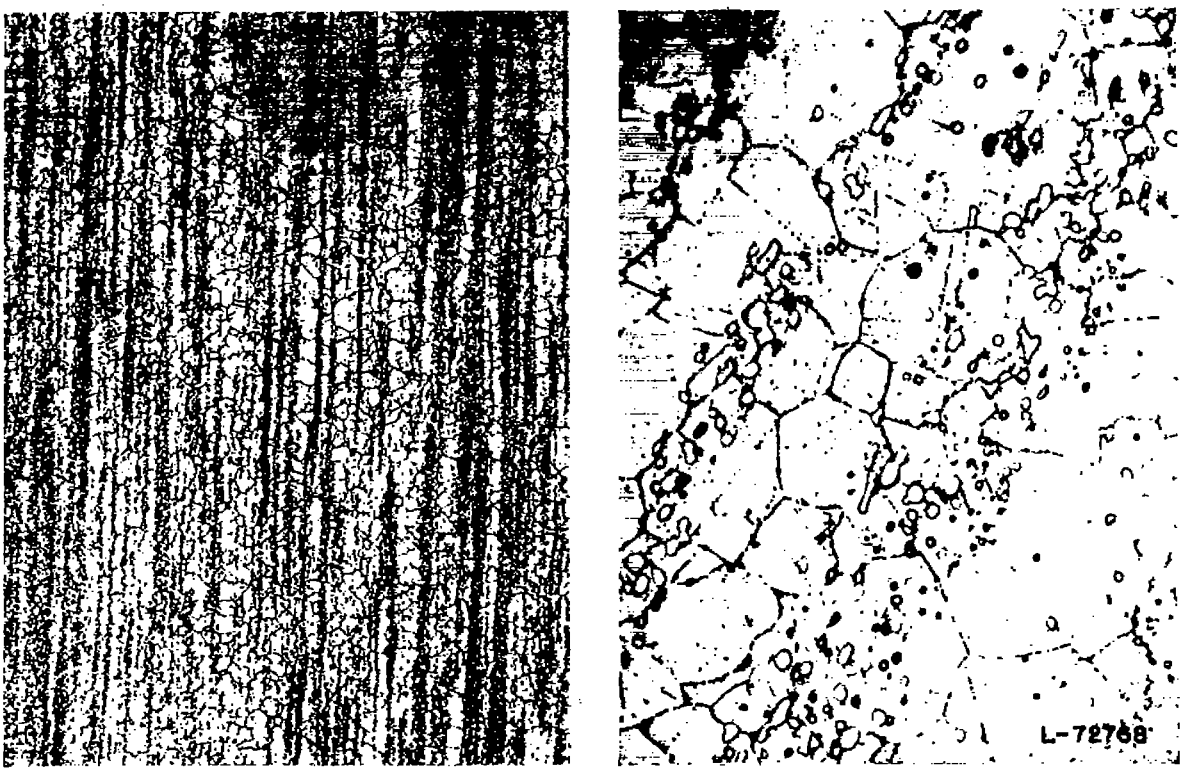
(d) Columbium, 6 percent (heat 50).

FIGURE 15.—Concluded.



100X 1000X

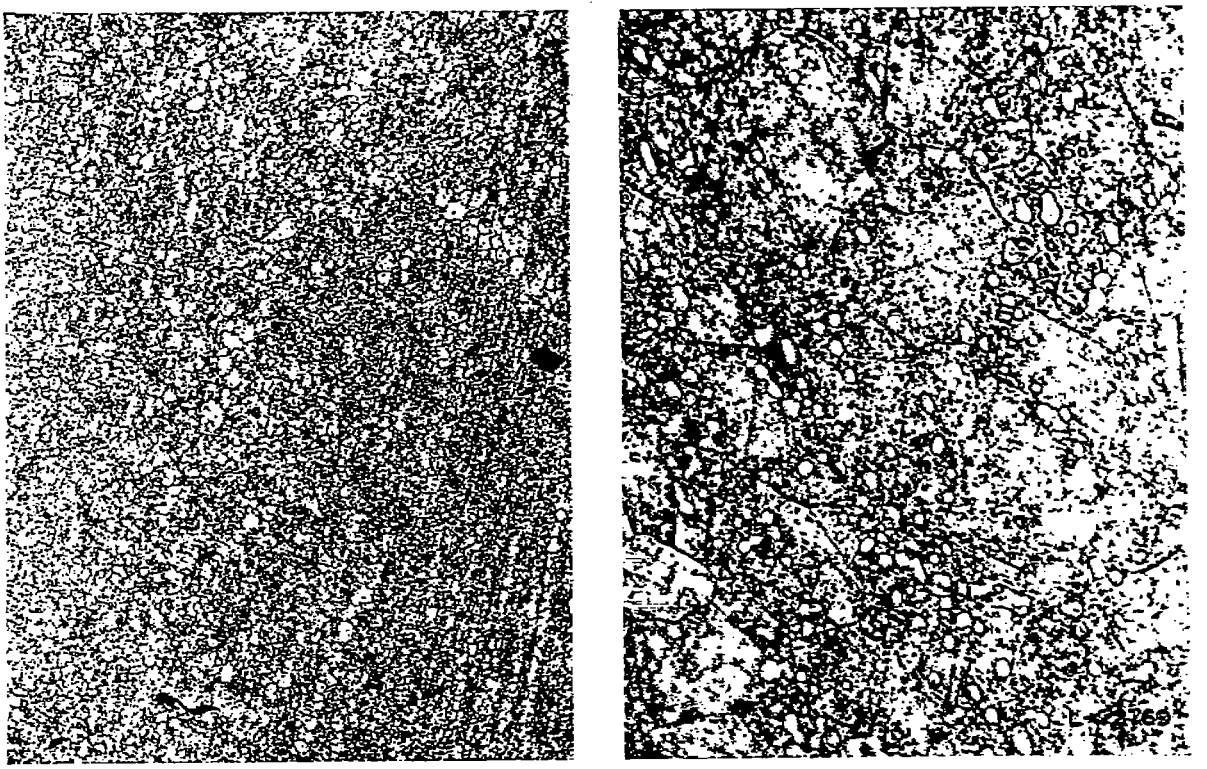
(a) Molybdenum, 0 percent; tungsten, 0 percent; columbium, 4 percent (heat 69).



100X 1000X

(b) Molybdenum, 2 percent; tungsten, 2 percent; columbium, 4 percent (heat 62).

FIGURE 16.—Influence of varying molybdenum and tungsten additions on microstructure of modified alloys containing 4 percent columbium. Treatment: 2200° F for 1 hour, water-quenched 1400° F for 24 hours.



100X 1000X

(c) Molybdenum, 4 percent; tungsten, 4 percent; columbium, 4 percent (heat 67).

FIGURE 16.—Concluded.

(2) 0Mo–0W–0Cb alloy:

Classification—I 1 z Cb

I—small amount of insoluble constituent

1—number 1 A. S. T. M. average grain size

z—large amount of precipitate near the grain boundaries

Cb—medium (b) amount of matrix precipitate on crystallographic planes (C)

The microstructural classifications are given in table VII for all the experimental alloys arranged for comparative purposes on the basis of the alloying element varied. For the alloys in which simultaneous variations were made in Mo, W, and Cb the classifications are repeated to show the influence of individual variations of these elements.

Influence of alloying elements on microstructure.—The effects on microstructure of variations of the elements will be given on the basis of the classification of table VII, supplemented by photomicrographs showing only the more pronounced effects observed.

Carbon: It is indicated in table VII and by the microstructures in figure 9 that as C was increased from 0.08 to 0.60 percent in the basic alloy the amount of insoluble constituent and matrix precipitate increased considerably and the grain size decreased, while the amount of grain boundary precipitate and the randomness of the matrix precipitate did not change. Figure 10 also shows that the amount of insoluble constituent was not reduced by heating the 0.60-percent-C alloy 10 hours at 2200° F over what it was after 1 hour.

The increase in the amount of soluble and insoluble constituents indicates that C is an important component in both the soluble and precipitating phases found in the basic alloy and its modifications.

Manganese: There was no apparent effect of Mn on microstructure.

Silicon: As will be shown later, Si was the only element which consistently lowered rupture strength over the complete composition range studied (from 0.5 percent in the basic alloy to 1.6 percent). The microstructures in figure 11 indicate that variation of Si did not change microstructural characteristics except for a smaller grain size for the high-Si alloy.

Chromium: Increasing Cr from 10 percent to the 20 percent of the basic alloy and to 30 percent drastically changed the microstructure as shown by the classification in table VII and the microstructures in figure 12. Increasing Cr caused the following changes: (1) The grain size became larger; (2) the amount of grain boundary precipitate increased between 10 and 20 percent Cr; (3) the matrix precipitate became heavier, especially between 20 and 30 percent Cr; and (4) for the 30-percent-Cr alloy the matrix precipitation occurred on the crystallographic planes rather than having a random pattern as for the lower-Cr alloys.

Nickel: The only pronounced structural variation for the series of Ni alloys was the ferrite-sigma-type phase which was present in the 0-percent-Ni alloy (see fig. 13). In the solution-treated alloy this phase was free from precipitate and the alloy was magnetic. In the aged condition a large amount of precipitation occurred in this phase and the alloy was nonmagnetic, signifying the transformation from the magnetic ferrite-type phase to the nonmagnetic sigma phase.

Upon rupture testing at 1200° F the magnetism increased with testing time, indicating some reversion to the ferrite-type phase. The 0-percent-Ni alloy also had less grain boundary precipitate and less precipitate in the austenite matrix than the higher-Ni alloys. None of the ferrite-type phase was observed in the alloy containing 10 percent Ni or in any of the other alloys studied in this investigation. Alloys containing 10 and 30 percent Ni were very similar in structure to the 20-percent-Ni basic alloy.

Cobalt: Varying Co from 0 to 32 percent produced two marked effects (see table VII and fig. 14). The 0-percent-Co alloy had a large amount of random matrix precipitation in the aged condition. The 10-percent-Co alloy resembled the basic 20-percent-Co alloy having only a medium amount of matrix precipitate. Increasing Co from 20 to 32 percent produced an alloy in which marked germination occurred at the 2200° F solution-treating temperature, resulting in several very massive grains surrounded by grains of normal size across the bar.

Another effect of Co on the structure occurred during rupture testing. Much additional matrix precipitation occurred in the 0-percent-Co alloy during testing; some also occurred in the 10-percent-Co alloy, and there was very little additional precipitate in the 20- and 32-percent alloys.

There was no evidence that Co changed the amount of the insoluble constituent or the grain boundary precipitate in the alloys.

Nitrogen: There was no appreciable influence from N varied from 0.004 to 0.18 percent.

Molybdenum: There were 10 modifications of alloys in which Mo was varied between 0 and 4 percent with 10 different constant ratios of W and Cb.

In the basic composition Mo was varied from 0 to 7 percent (alloys 32 to 36 in table VII). The only apparent effect of Mo was the larger grains at both the lower and the higher percentages.

For the nine other alloy modifications (alloys 43, 45, 46, 53 to 73, and 82 to 84) increases in Mo to 2 and 4 from

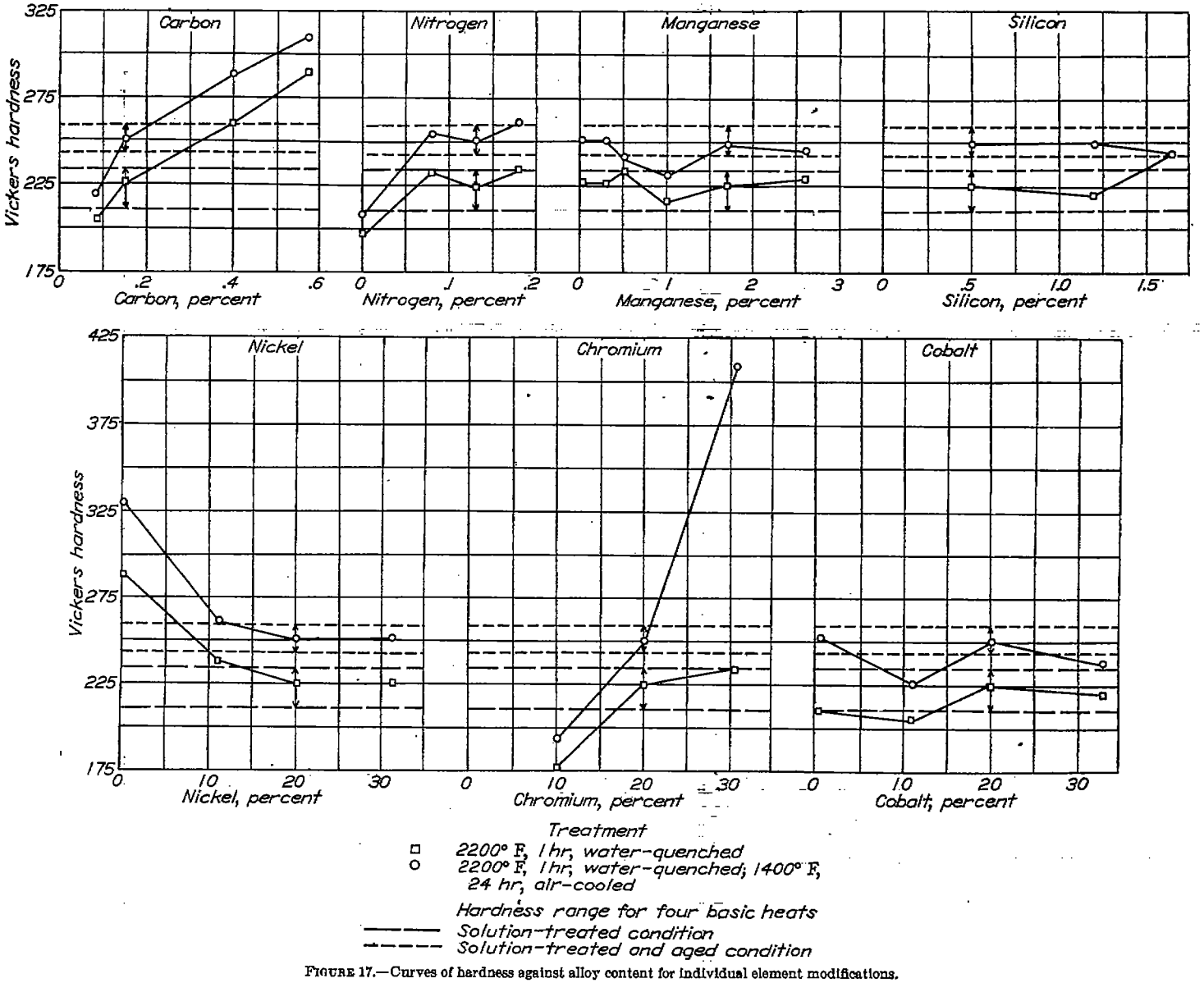


FIGURE 17.—Curves of hardness against alloy content for individual element modifications.

0 percent produced only very slight changes in structure except in the alloys containing Cb (particularly 4 percent Cb) in which case the grain size was refined and the amount of matrix precipitation during aging was increased.

Major structural changes between these alloy modifications were the result of variation in Cb.

Tungsten: In the 10 alloy modifications in which W was varied systematically (alloys 37 to 40, 43, 45, 46, 53 to 73, and 82 to 84 in table VII) similar observations to those for the influence of Mo were made. Increases in W slightly refined the grains and increased the matrix precipitation to a small degree in some cases.

Columbium: As shown in table VII for the 10 alloy modifications with variable Cb, this element had a marked influence on all the structural classification variables. A typical example of microstructural changes occurring with Cb additions from 0 to 6 percent to the basic composition is shown in figure 15.

Increasing Cb from 0 to the 1 percent of the basic alloy produced the following structural changes: (1) Increased the insoluble constituent from very little to an appreciable amount; (2) drastically reduced grain size; (3) decreased the concentration of grain boundary precipitation during aging; (4) changed the mode of matrix precipitation from internal crystallographic planes to a random pattern; and (5) decreased the amount of matrix precipitation during aging.

Increasing Cb from the 1 percent of the basic alloy to 4 percent continued to increase markedly the amount of insoluble constituent, to further gradually reduce grain size, and not to alter appreciably the grain boundary concentration effects or the randomness of precipitation while increasing the amount of matrix precipitation during aging in alloys containing four or more percent of Mo or W.

While Cb did produce major changes in microstructure, it is noted that for the alloys arranged in table VII with Cb as the systematic variable the presence of either or both Mo or W in the alloys increased the tendencies for these changes. This is shown in figure 16 for 4-percent-Cb alloys containing increasing Mo and W contents.

Summary of influence of alloying elements on microstructure.—A summary of the effects of alloying elements on tendencies for microstructural changes is given in table VIII.

Two elements producing major microstructural changes were Cb and C. Both elements increased the amounts of insoluble constituent and aging precipitate and refined the grain size of material solution-treated at 2200° F. In addition, Cb changed the mode of matrix precipitation from

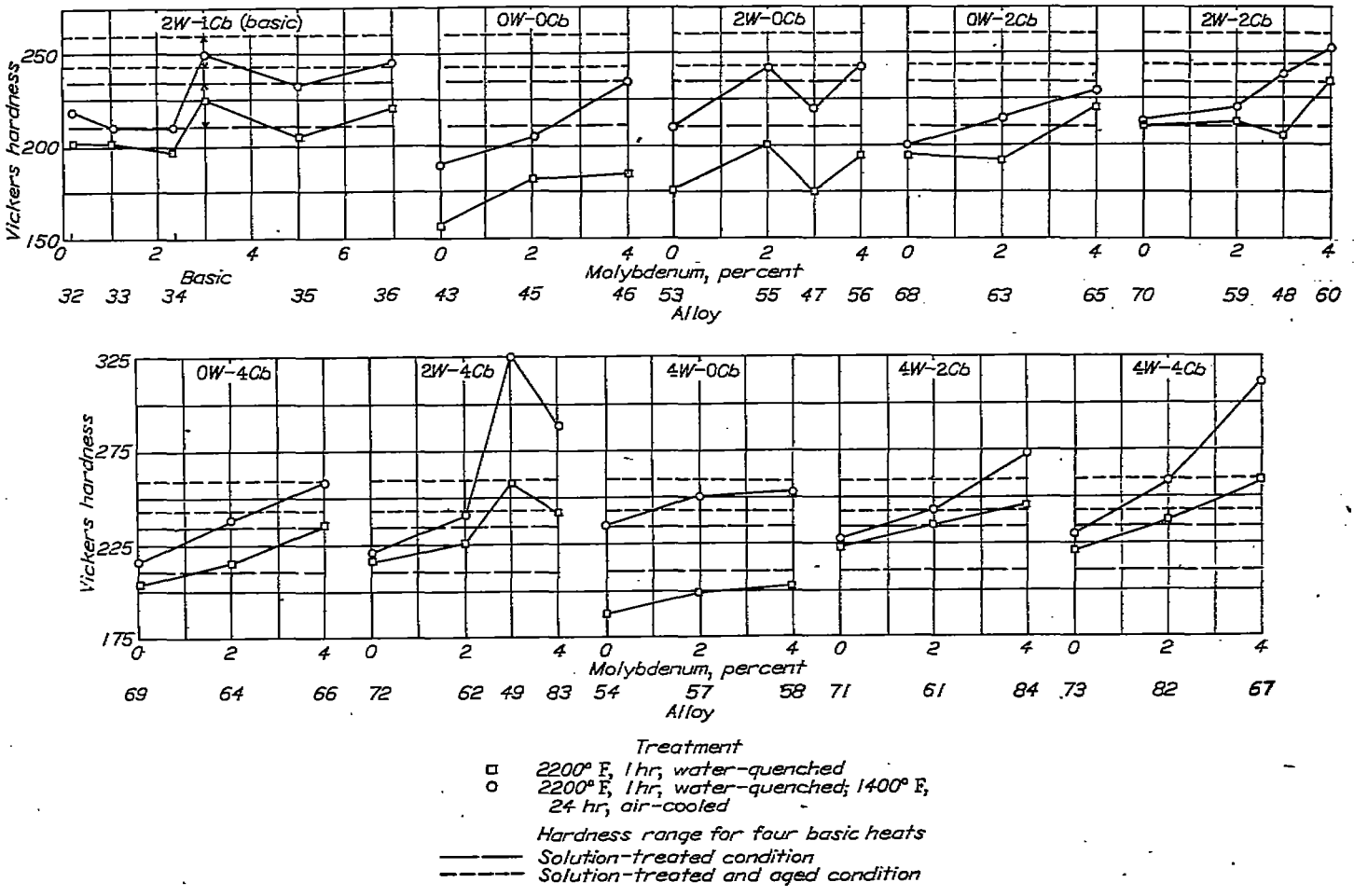


FIGURE 18.—Curves of hardness against molybdenum content for 10 tungsten-columbium modifications.

preferred, when no Cb was present, to random, when the 1 percent of the basic alloy or more was present.

Additions of Mo and W produced little change in microstructure except in alloys containing 4 percent Cb in which case these elements decreased the grain size and increased matrix precipitation.

Between 20 and 30 percent of Cr a pronounced increase occurred in the amount of aging precipitate and the mode of precipitation changed from random to preferred.

Grain size was mildly increased and the amount of aging precipitate decreased by Co.

Additions of Ni (between 10 and 30 percent) produced only minor changes in structure. The 0-percent-Ni alloy contained a ferrite-sigma type of phase which disappeared when Ni was raised to 10 percent.

The only effect of Si, within the limits studied, was to refine grain size slightly. No discernible change in structure was produced by Mn and N.

HARDNESS

Vickers hardness tests at room temperature were made on metallographic specimens of all the experimental alloys in the solution-treated (2200° F, 1 hr, water-quenched) and the aged (1400° F, 24 hr, air-cooled) conditions. Preliminary hardness surveys on several alloys indicated that the forged bars had uniform hardnesses both in the lengthwise and the crosswise directions.

Hardness test results, representing the average of at least four tests on each sample, are given in table IX and are plotted against composition variables in figures 17 to 20.

There was fair agreement between the hardnesses of five heats of the basic alloy. Additions of C, N, Cr, Mo, W, and Cb tended to increase hardness in both conditions; Ni, particularly from 0 to 10 percent, lowered hardness; and Mn, Si, and Co did not appreciably affect hardness. Perhaps an exception to this was the effect observed for the 1.6-percent-Si alloy, which was the only one of all the alloys studied which did not show hardening as a result of aging. The addition of Cr from 20 to 30 percent markedly increased the age-hardening characteristics of the alloy.

Figure 18 shows the influence of Mo for 10 W–Cb modifications. Additions of Mo, while tending to increase hardness, did not appreciably increase the age-hardening tendency except for the modifications containing more than 4 percent total of W plus Cb. For the 10 Mo–Cb modifications (fig. 19) the effect of W additions was similar to that observed for Mo, although there was less tendency for increasing age hardening with higher alloy contents. In both figures 18 and 19 it is seen that there was a greater difference between the hardness in the solution-treated and the aged conditions

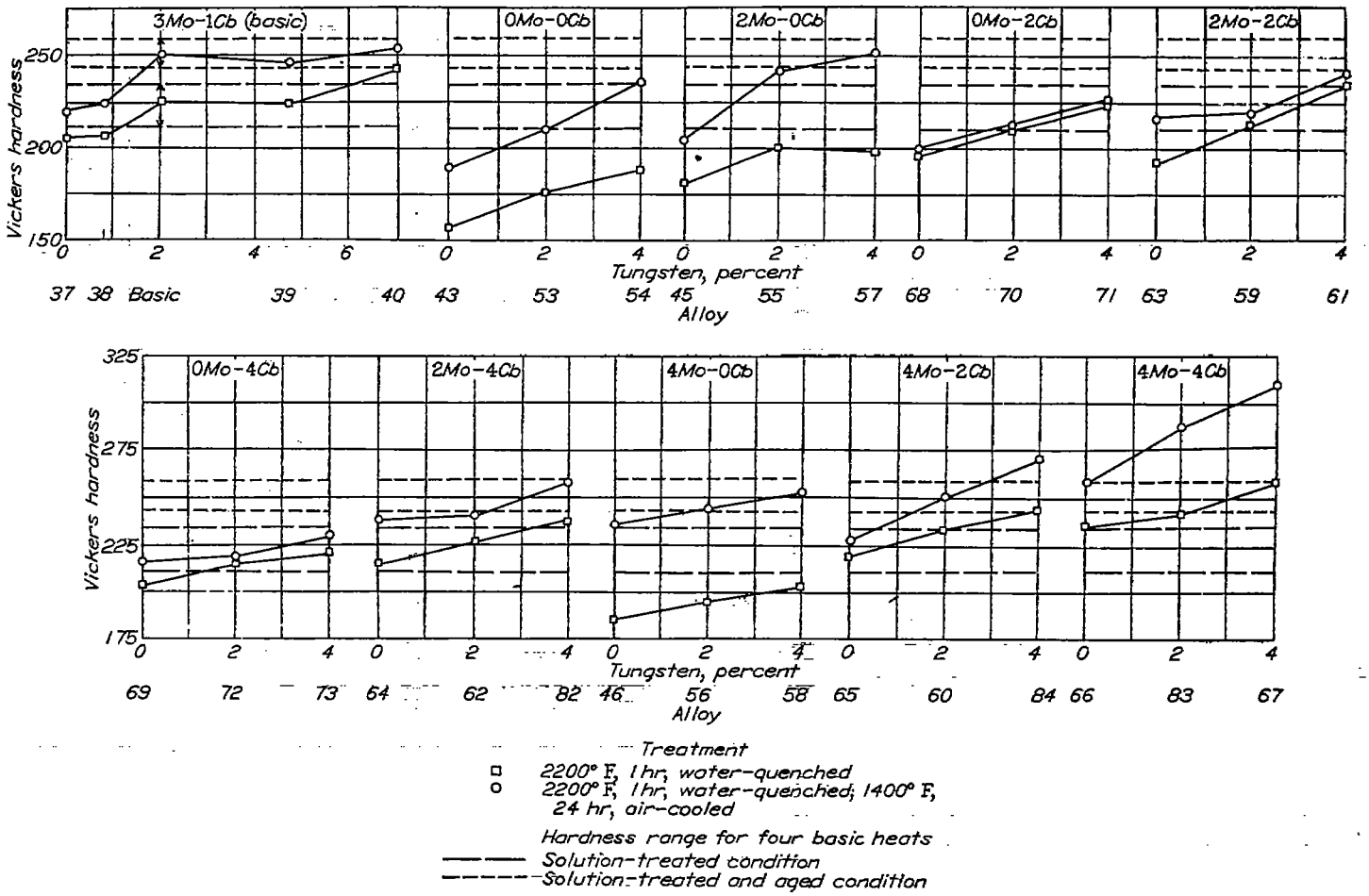


FIGURE 19.—Curves of hardness against tungsten content for 10 molybdenum-columbium modifications.

for the modifications not containing Cb than for the modifications containing Cb. This effect is shown again in figure 20 in that Cb additions from 0 to 2 percent to the 10 Mo–W modifications narrowed the age-hardening range. Additions of Cb from 2 to 4 percent tended to broaden the age-hardening range for modifications containing the larger amounts of Mo and W.

It is recognized that these hardness results are not a true evaluation of the comparative aging effects caused by composition variables. Such an evaluation would necessarily require aging-time-temperature data.

INFLUENCE OF CHEMICAL-COMPOSITION VARIABLES ON RUPTURE TEST CHARACTERISTICS AT 1200° F

The results of rupture tests at 1200° F for all the alloys studied are given in table X. Included in this table are the times for rupture, elongations, reductions of area, and minimum creep rates obtained from the curves of elongation against time for the individual tests. Also included are 100- and 1000-hour rupture strengths obtained from double-logarithmic plots of stress against rupture time (an example is shown in fig. 5); estimated elongation to rupture in 100 hours; stresses for creep rates of 0.1 and 0.01 percent per hour obtained from double-logarithmic plots of stress against minimum creep rates from the rupture tests (an example is shown in fig. 6); and minimum creep rates at 40,000 psi. The alloys are listed in table X in the approximate order of the element varied in the basic analysis.

Rupture tests of sufficient number and duration were conducted to establish the 100-hour rupture strengths. The 1000-hour rupture strengths were obtained by extrapolation of the double-logarithmic curves of stress against rupture time. Unless the maximum-time rupture test was longer than 500 hours, however, the 1000-hour strengths are listed in table X as estimated and are indicated as such on the curves of strength against chemical composition.

In this evaluation of the influence of variations in chemical composition on the properties most emphasis has been placed on rupture properties at 1200° F. Because the creep data were obtained from the rupture tests, it is emphasized that the rates of deformation were much higher than those usually associated with reported "creep strength."

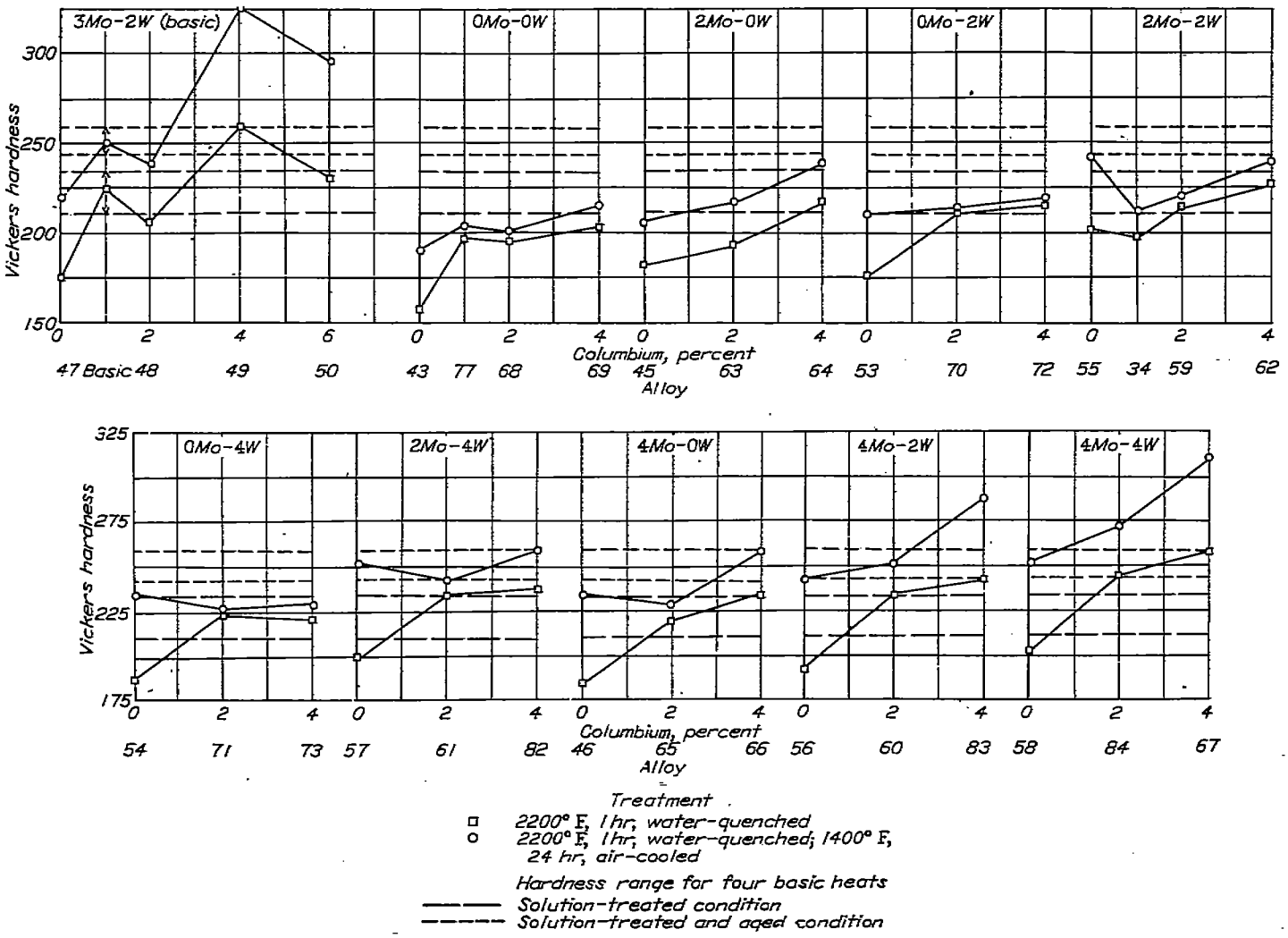


FIGURE 20.—Curves of hardness against columbium content for 10 molybdenum-tungsten modifications.

100-HOUR RUPTURE PROPERTIES AT 1200° F OF MODIFIED ALLOYS

The 100-hour rupture strengths and elongations for all of the alloys are arranged in order of increasing 100-hour rupture strengths in figure 21. The ranges in properties for the basic alloy heats, forged both as squares and as rounds, are indicated to show the significant variations in properties. The following observations are made:

(1) The over-all 100-hour rupture strength range was from 26,000 to 52,000 psi. The lowest-strength alloy was the one which did not contain Mo, W, and Cb. The two highest-strength alloys were modifications of the basic alloy, one containing 30 percent Cr, the other 7 percent W.

(2) Six alloys had rupture strengths above the range of the basic alloy forged to round bars. None of the alloys, however, were above the range for the basic alloy forged to squares where the practice was not so well controlled.

(3) It appears that all of the elements can be varied individually over relatively wide ranges without appreciably altering rupture strengths. It will be shown, however, that in most cases rupture strengths varied consistently with systematic variations in composition.

(4) Alloys having strengths near the lower end of the range were those containing the smallest amounts of Mo, W, or Cb added separately or two at a time. Additions of greater amounts of these elements generally yielded rupture strengths which were closer to the strength range of the basic alloy.

(5) Adding 2 percent of either Mo, W, or Cb to the lowest-strength alloy increased the 100-hour rupture strength in the order of 10,000 psi or more.

(6) Strengths in the order of those of the basic alloy were obtained without the presence of Cb. Strengths almost as high were obtained without the presence of Mo or of W.

(7) Low Cr, Co, or Ni and high Si resulted in lower strengths than the range for the basic heats.

(8) Elongation at rupture in 100 hours ranged from 5 to 40 percent.

(9) There was no relationship apparent between rupture elongation and strength. The omission of Cb resulted in consistently low elongation. More Cr or Co than the 20 percent of the basic alloy also lowered elongation.

INDIVIDUAL VARIATIONS OF ELEMENTS

The influence on rupture test characteristics of systematic variations of the individual elements, shown in figures 22 to 31, was as follows:

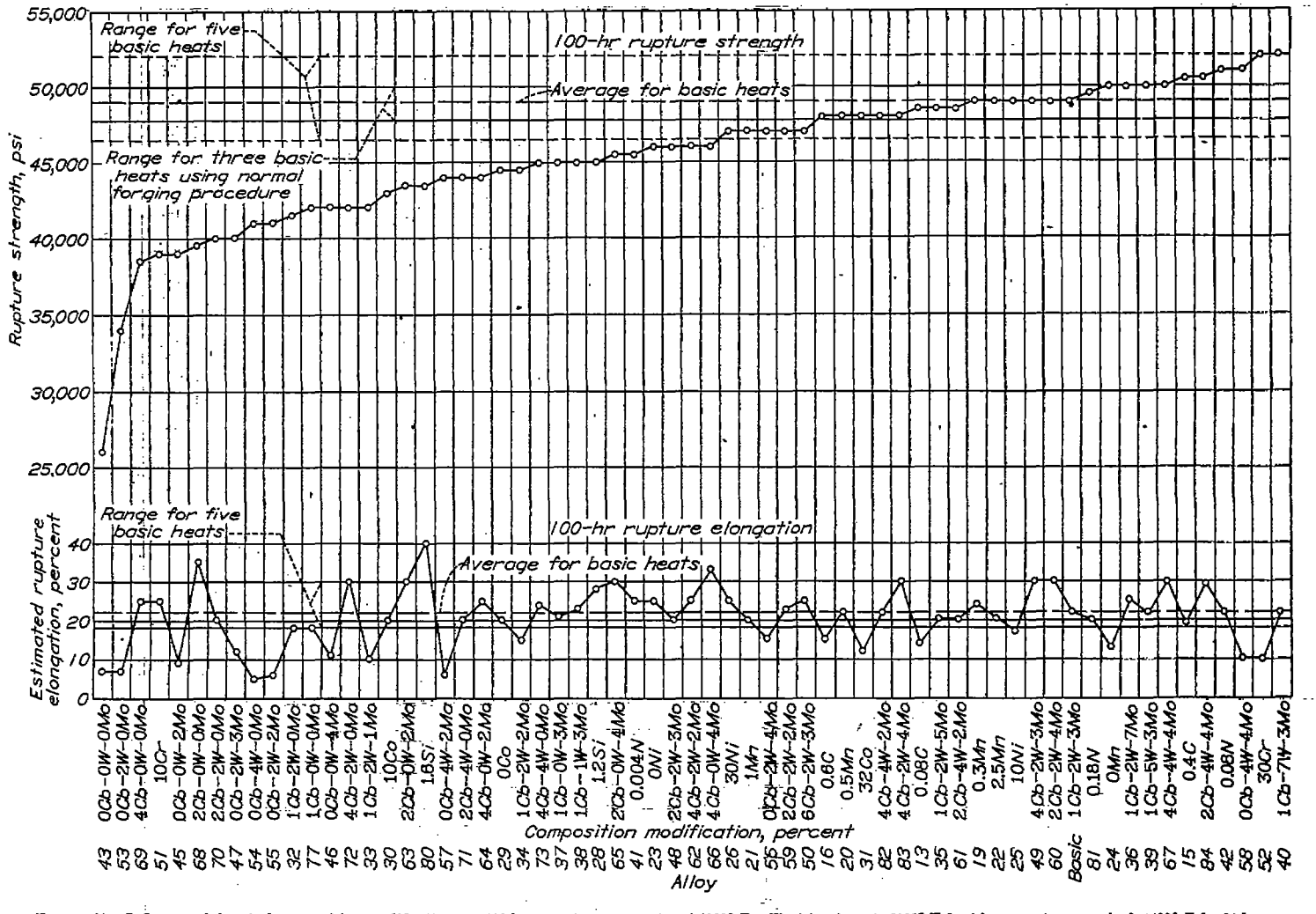


FIGURE 21.—Influence of chemical-composition modifications on 100-hour rupture properties at 1200° F. Heat treatment: 2200° F for 1 hour, water-quenched; 1400° F for 24 hours.

Carbon.—The effect of C, varied from 0.08 to 0.60 percent, was only slight. All the rupture strengths fell within close proximity to those of the basic alloy (see fig. 22). The variable C heats were forged as squares and are compared with the property ranges found for similarly forged basic heats. The lowest and the highest C heats had rupture elongations below normal.

Nitrogen.—Variations of N, from 0.004 to 0.18 percent, tended to increase strength somewhat (see fig. 23). The alloy containing 0.004 percent N had strengths which were slightly below those of the basic 0.12-percent-N alloy while the 0.08-percent-N alloy was slightly stronger.

Manganese.—Figure 24 shows that Mn variations from 0 to 2.5 percent had no significant influence on rupture

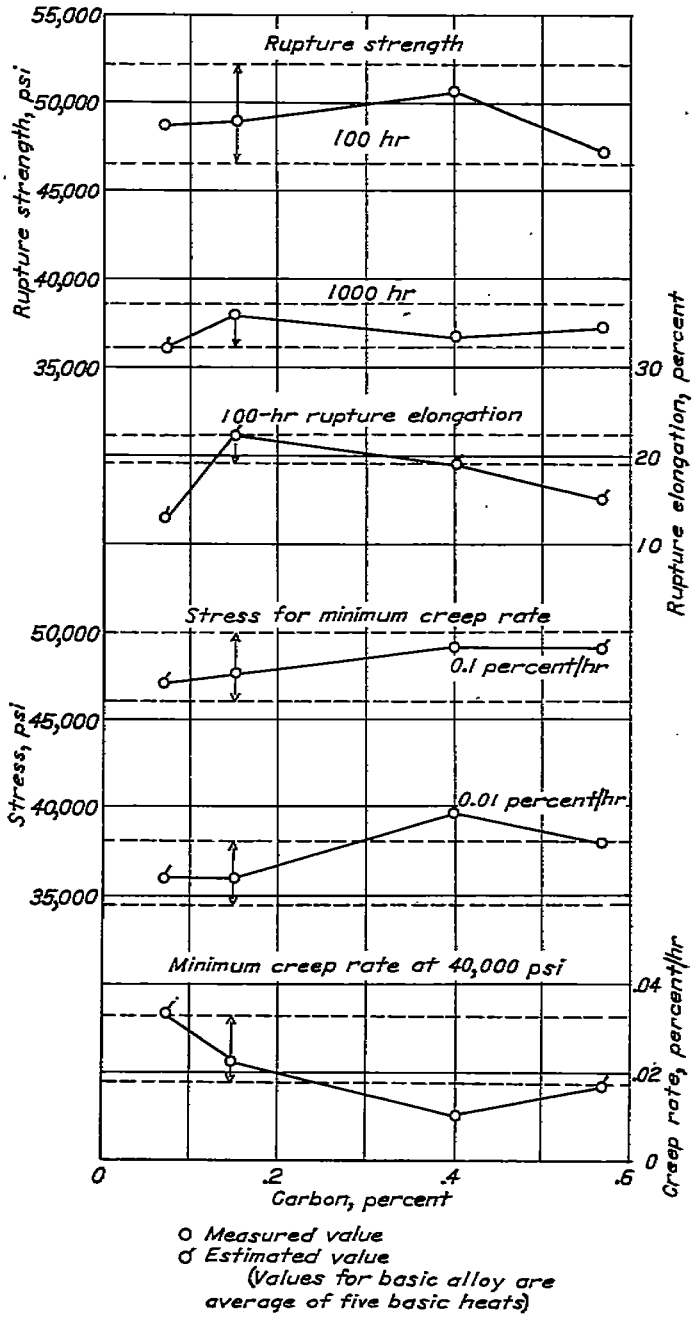


FIGURE 22.—Curves of rupture test data at 1200° F against carbon content of basic alloy. Horizontal dashed lines indicate property range for five basic heats.

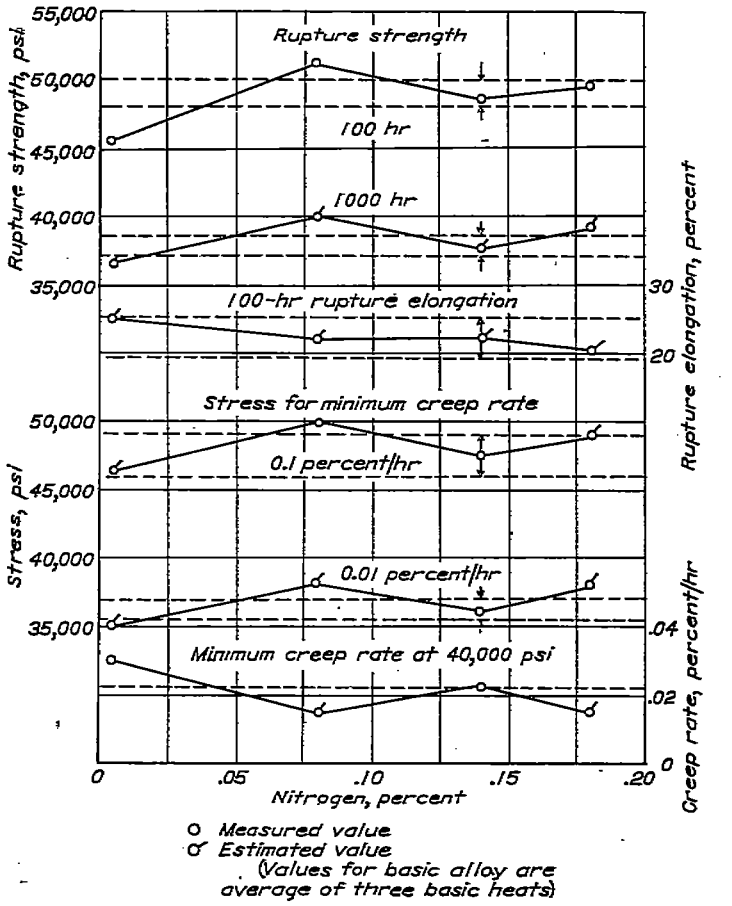


FIGURE 23.—Curves of rupture test data at 1200° F against nitrogen content of basic alloy. Horizontal dashed lines indicate property range for three basic heats.

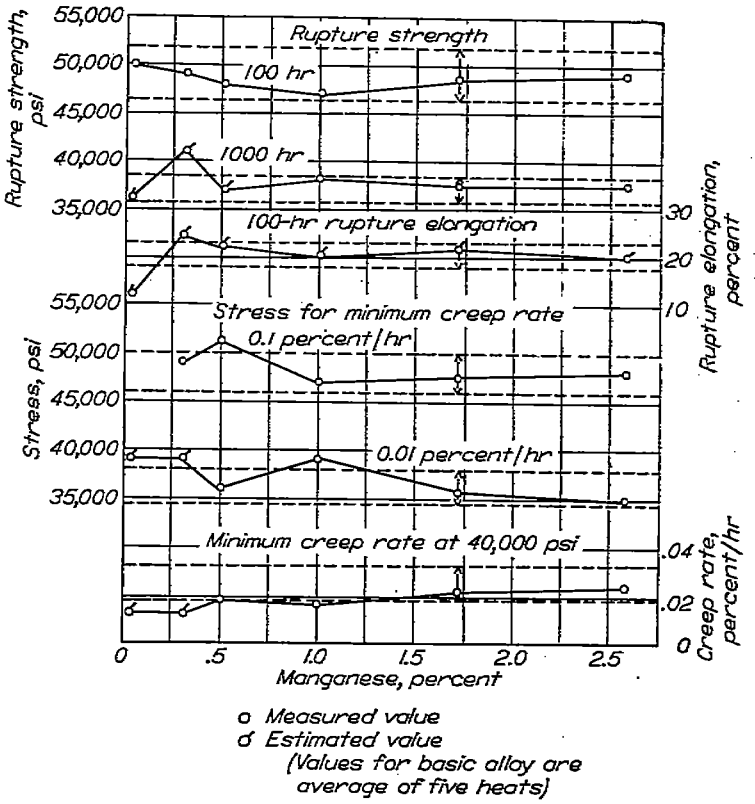


FIGURE 24.—Curves of rupture test data at 1200° F against manganese content of basic alloy. Horizontal dashed lines indicate property range for five basic heats.

properties except a somewhat lower elongation for the low-Mn heat.

Silicon.—Figure 25 shows that increasing Si from the 0.5 percent of the basic alloy to 1.6 percent produced a marked lowering of rupture strength and an increase in elongation. The stresses at constant creep rates were lowered and creep rates at 40,000 psi were noticeably raised in agreement with trends in rupture properties. This was the only case in which the addition of an element consistently lowered strength over the complete composition range studied.

Chromium.—The influence of Cr variations from 10 to 30 percent is shown in figure 26. The 100-hour rupture strengths were increased from 39,000 to 52,000 psi while rupture elongation decreased from 25 to 10 percent. Similar marked

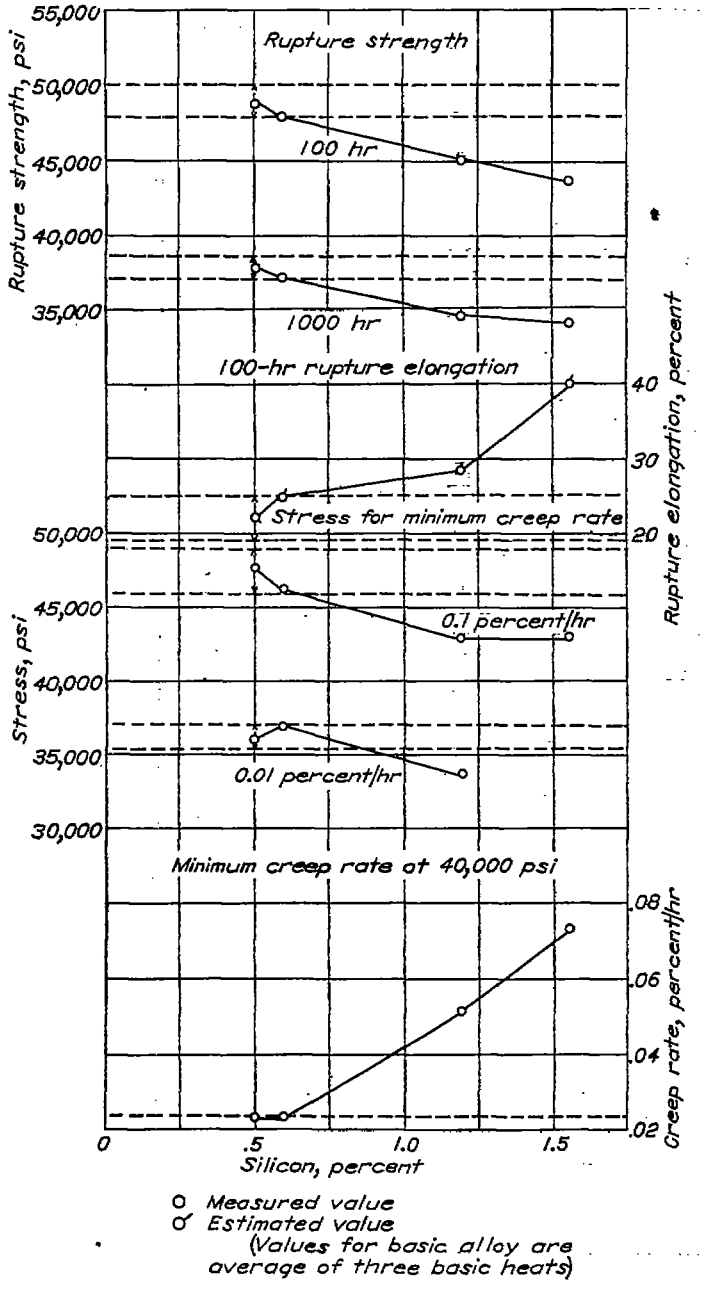


FIGURE 25.—Curves of rupture test data at 1200° F against silicon content of basic alloy. Horizontal dashed lines indicate property range for three basic heats.

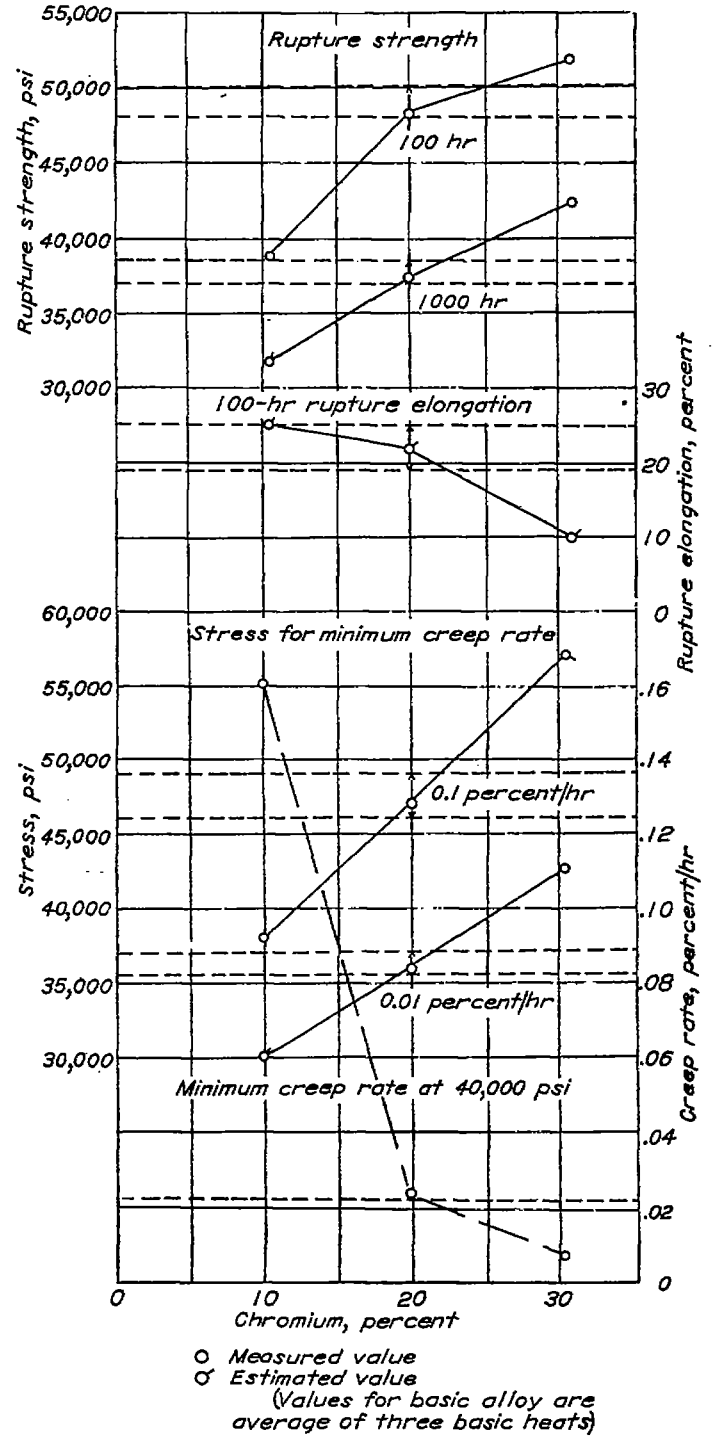


FIGURE 26.—Curves of rupture test data at 1200° F against chromium content of basic alloy. Horizontal dashed lines indicate property range for three basic heats.

increases occurred in 1000-hour rupture strengths and stresses at constant creep rates while the creep rate at 40,000 psi was drastically reduced, particularly between 10 and 20 percent Cr.

Nickel.—Additions of Ni from 0 to 30 percent had little influence on properties as shown in figure 27. The 0- and 30-percent-Ni alloys had slightly lower rupture strengths than the range for the basic 20-percent-Ni heats, producing an apparent maximum in strength between 10 and 20 per-

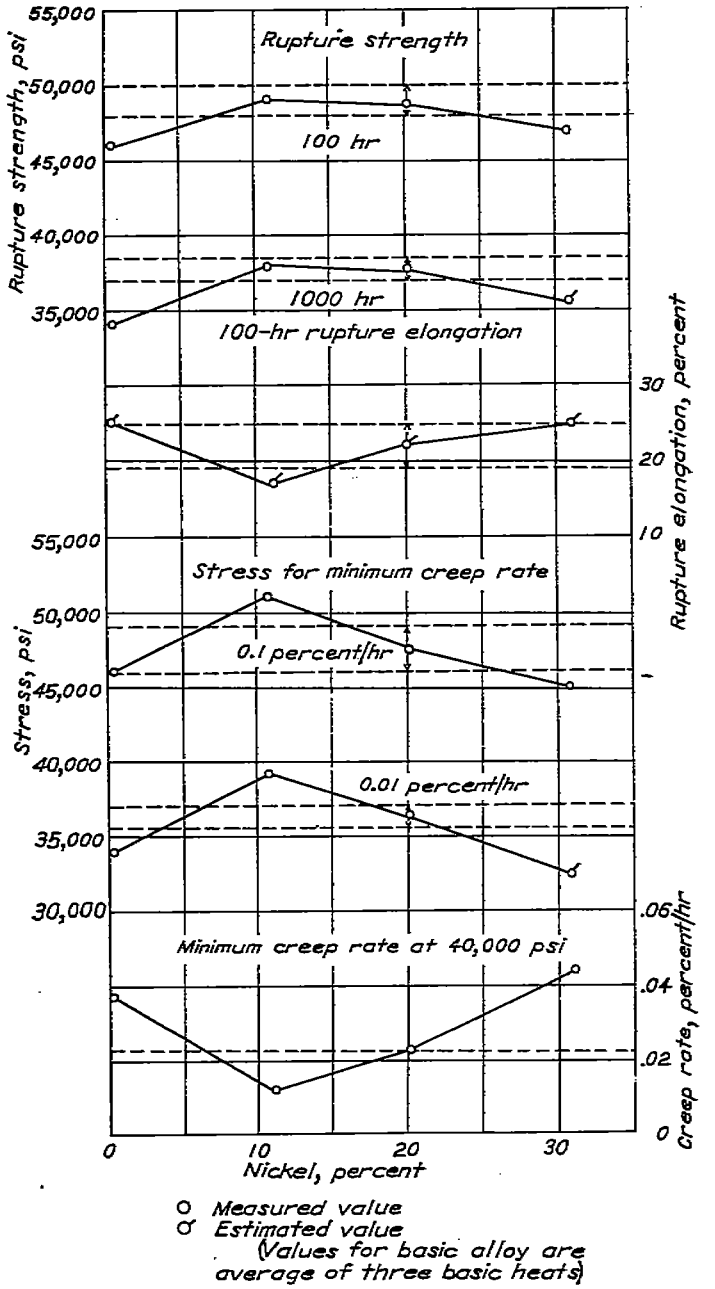


FIGURE 27.—Curves of rupture test data at 1200° F against nickel content of basic alloy. Horizontal dashed lines indicate property range for three basic heats.

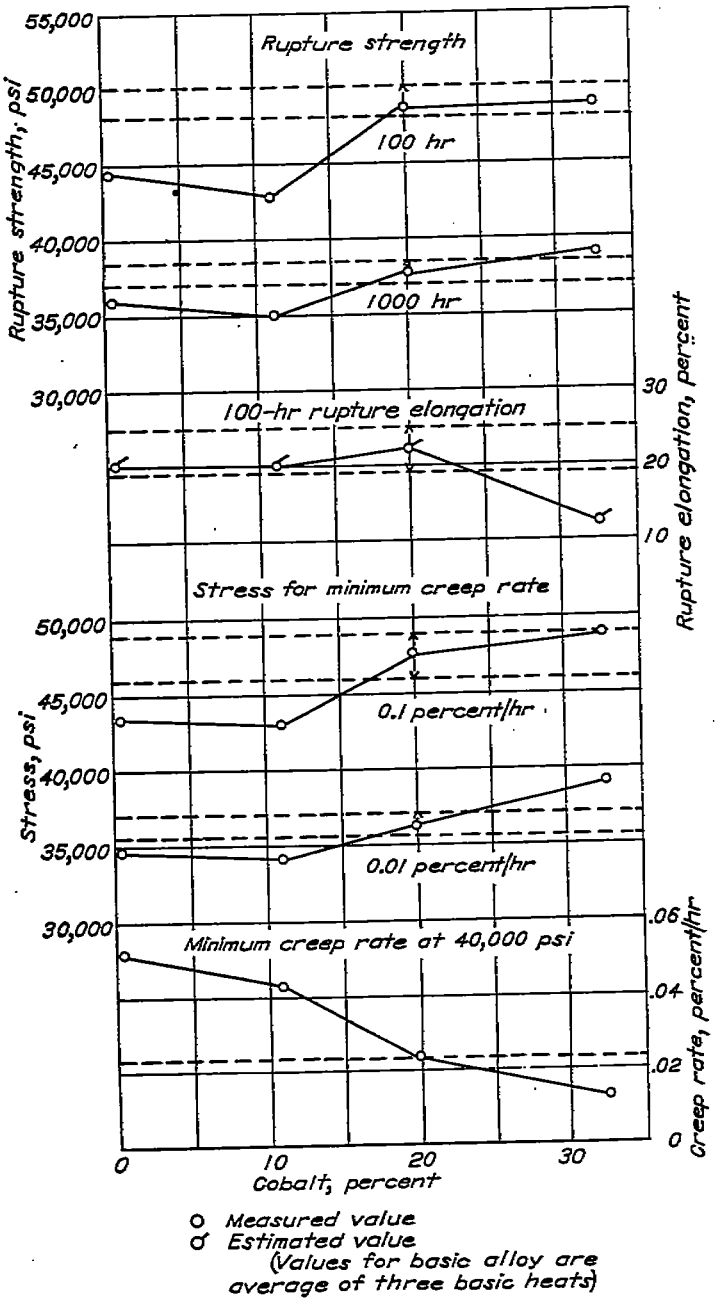


FIGURE 28.—Curves of rupture test data at 1200° F against cobalt content of basic alloy. Horizontal dashed lines indicate property range for three basic heats.

cent Ni. A similar slight trend was noted in stresses at constant creep rates while the creep rate at 40,000 psi decreased and then increased with increasing Ni, following the trend of the change in elongation at fracture in 100 hours.

Cobalt.—Figure 28 shows the influence of Co variations from 0 to 32 percent. The 0- and 10-percent-Co alloys had lower rupture strengths than normal with little effect on elongation. The 20- and 32-percent-Co alloys had equal 100-hour rupture strengths at a higher level than those with lower Co. The 1000-hour rupture strengths and stresses at constant creep rates were improved, while the elongation and creep rate were lowered, when Co was increased from the 20 percent of the basic alloy to 32 percent.

Molybdenum.—Additions of Mo from 0 to 7 percent improved rupture and creep properties as shown by figure 29. The most marked improvement in rupture strength from Mo was from additions of 1 to 3 percent.

Tungsten.—Figure 30 shows that the improvement in rupture strength with additions of W from 0 to 7 percent was gradual over the entire range, this addition increasing 100-hour strength from 45,000 to 52,000 psi. The stresses at constant creep rates showed a similar trend to rupture strengths while rupture elongation was not at all affected by W.

Columbium.—Figure 31 shows that increasing Cb from 0 to the 1 percent of the basic alloy increased the 100-hour rupture strength from 40,000 to 48,000 psi. Additions of Cb

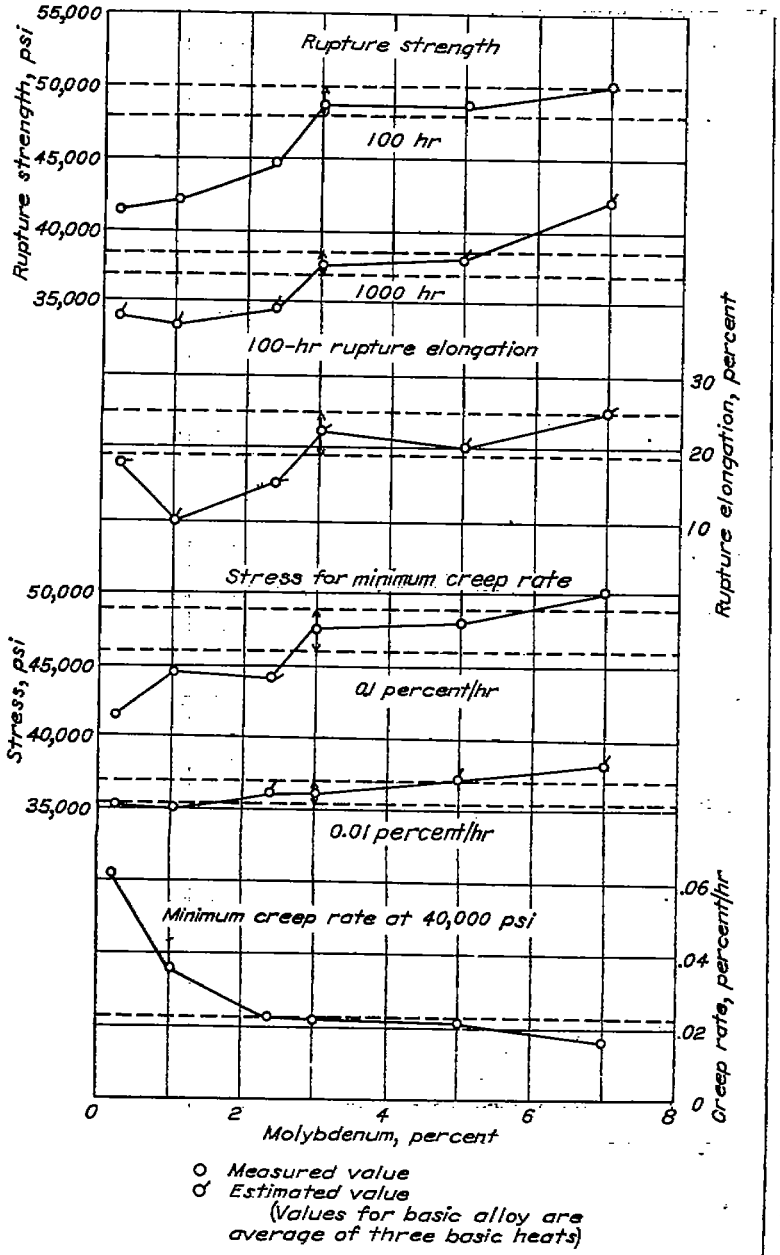


FIGURE 29.—Curves of rupture test data at 1200° F against molybdenum content of basic alloy. Horizontal dashed lines indicate property range for three basic heats.

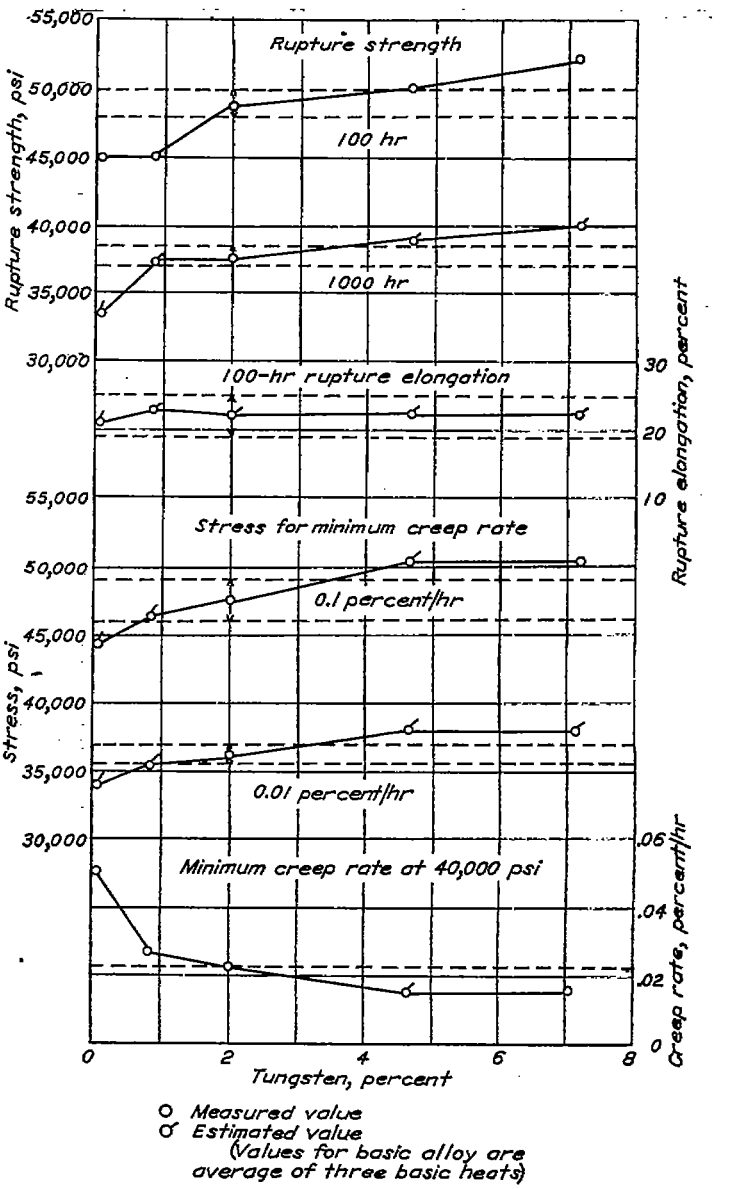


FIGURE 30.—Curves of rupture test data at 1200° F against tungsten content of basic alloy. Horizontal dashed lines indicate property range for three basic heats.

from 1 to 6 percent produced no further improvement in strength. A similar trend was shown by creep properties. However, the increase of 44,500 to 47,500 psi in the stress for a minimum creep rate of 0.1 percent per hour between 0 and 1 percent Cb was not of the same magnitude as the rupture strength increase. Rupture test elongation was raised from 12 to 22 percent by the addition of 1 percent Cb and tended to increase further with higher Cb.

SIMULTANEOUS VARIATIONS OF MOLYBDENUM, TUNGSTEN, AND COLUMBIUM

In addition to the individual variations of all the elements the effects of simultaneous variations of Mo, W, and Cb in steps of 2 percent from 0 to 4 percent were evaluated. This made possible curves showing the influence on properties of systematic variations of one of these elements for 10 different constant ratios of the other two elements.

Molybdenum.—The influence of the Mo variations from 0 to 4 percent on rupture test characteristics of alloy modifications, including the basic alloy, with 10 different constant ratios of W and Cb is shown in figure 32. Curves comparing the relative effect of Mo on the 100-hour rupture strength of the 10 W-Cb modifications are shown in figure 33.

Increasing Mo from 0 to 4 percent tended continuously to increase the 100-hour rupture strength for all the ratios of W and Cb. The relative effect was greatest for the modifications containing neither W nor Cb, the strength increase being from 26,000 to 42,000 psi. In alloys containing W or Cb the strengthening effect of Mo was greatest for the alloys which contained 2 and 4 percent of W and no Cb. This strengthening effect was less for alloys containing 2 and 4 percent Cb and no W and became even less for those containing both Cb and W which had a higher level of initial strength.

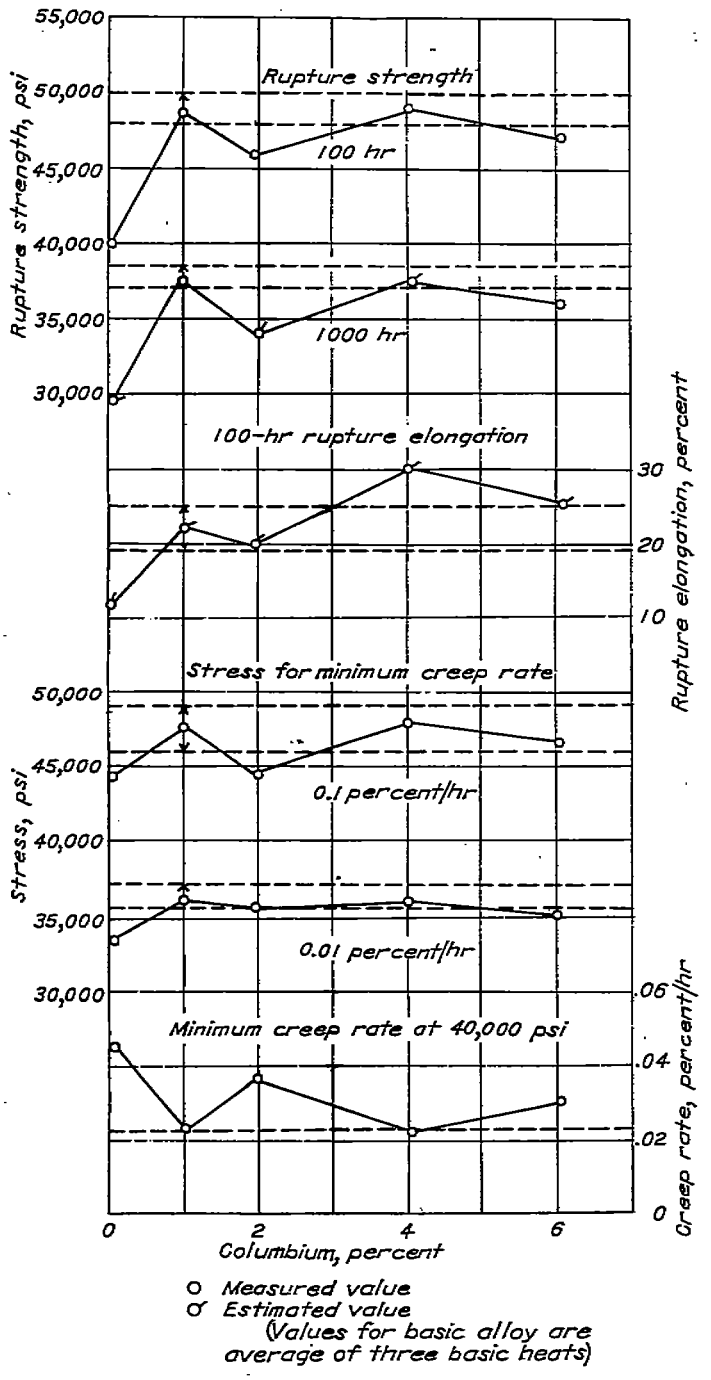


FIGURE 31.—Curves of rupture test data at 1200° F against columbium content of basic alloy. Horizontal dashed lines indicate property range for three basic heats.

The 1000-hour-strength trends followed those of the 100-hour strengths. There was a slight tendency for increasing Mo to widen the difference between the 100- and 1000-hour rupture strengths in the modifications containing more than 6 percent total of W plus Cb.

In general Mo tended to improve the 100-hour rupture elongation. It is noted that for the alloys in which Cb was absent the elongation was quite low (5 to 15 percent) as compared with the elongations above 20 percent of the alloys containing 2 and 4 percent Cb.

The stresses causing a minimum creep rate of 0.1 percent per hour followed the trends and were of the same order of magnitude as the 100-hour rupture strengths. The trends and magnitudes of stresses causing a creep rate of 0.01 percent per hour were somewhat similar to those of the 1000-hour rupture strengths. Creep stresses were consistently higher than rupture strength, however, for the 0-percent-Cb alloys having low rupture elongation and were somewhat lower than rupture strength for the alloys with higher elongation.

Tungsten.—By rearranging the data for alloys involving simultaneous variations of Mo, W, and Cb figures 34 and 35 were obtained. These show the influence of W variations from 0 to 4 percent on rupture test characteristics of alloy modifications with 10 different constant ratios of Mo and Cb.

Additions of W produced an almost linear increase in 100-hour rupture strength. The greatest improvement, 26,000 to 41,000 psi, was for the 0Mo–0Cb modifications, with the relative improvements tapering off for the alloys with higher Mo and Cb to about 4000 psi in the range 46,000 to 50,000 psi for the 4Mo–4Cb modifications. The 1000-hour rupture strengths, although only estimated in many cases, followed a similar, but not quite so pronounced, trend. The trends in creep properties were also, in general, the same as those for the rupture strengths with the additional effect of the level of elongation on the relation between rupture strength and stresses at constant creep rates.

There was a general tendency for W to lower the 100-hour rupture elongation very slightly. The low elongation of the alloys which did not contain Cb was again noted.

Columbium.—The data arranged to define the influence of Cb are shown in figures 36 and 37. The influence of Cb on rupture strengths was significantly different from the influences of Mo and W. Additions of Cb from 0 to 2 percent, in general, increased the 100-hour rupture strength, but there was no significant strength increase with greater additions. This finding, in addition to the observation of the influence of 1 percent as compared with that of 0 percent Cb on the strength of the basic alloy, led to the preparation of an additional alloy (77) containing 0Mo–0W–1Cb which fits into the 0Mo–0W alloy modifications as shown in figure 36. This alloy had as good or better strength properties than the 0Mo–0W–2Cb modification. The addition of Cb to the 4Mo–2W and 4Mo–4W modifications evidently had no effect or a slightly detrimental effect on rupture strength.

One or two percent of Cb markedly improved the rupture elongation. An additional slight improvement was realized with even higher Cb.

The stresses causing a creep rate of 0.1 percent per hour were higher than the 100-hour rupture strengths for 0-percent-Cb alloys but lower than the rupture strengths for alloys containing Cb. Thus the creep stress did not follow the increase in rupture strength with the additions of 1 or 2 percent Cb but either remained constant or decreased. It appeared that the marked increase in elongation with small Cb additions was the factor responsible for the improvement in rupture strength.

It can be concluded that, for the alloy modifications studied, additions of Cb are necessary to produce substantial ductility in the rupture test, but additions of more than 2, or probably 1, percent of Cb add nothing to the rupture properties of the alloys.

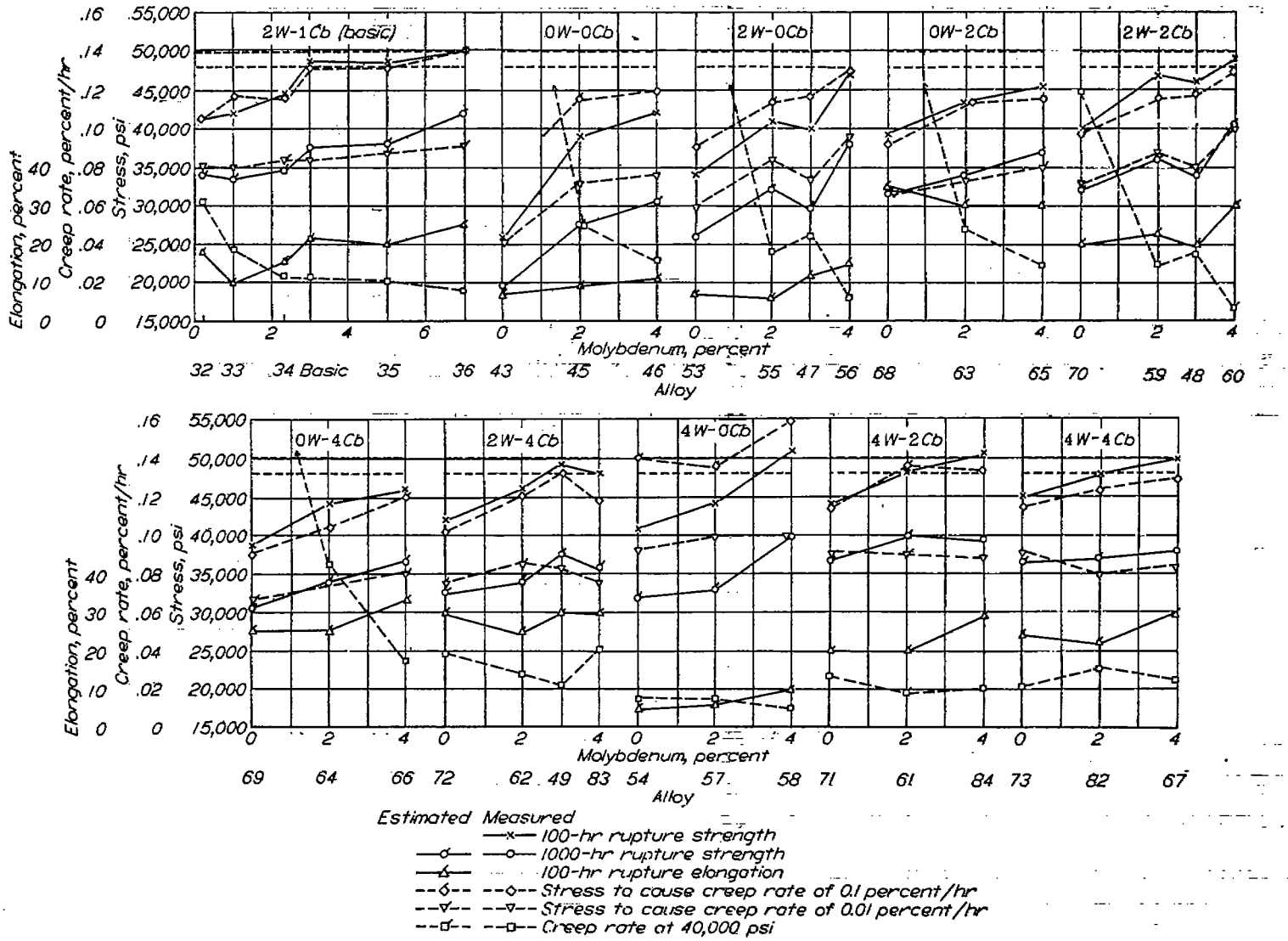


FIGURE 32.—Curves of rupture test data at 1200° F against molybdenum content for 10 tungsten-columbium modifications. Horizontal dashed lines indicate range of 100-hour rupture strength for three basic heats.

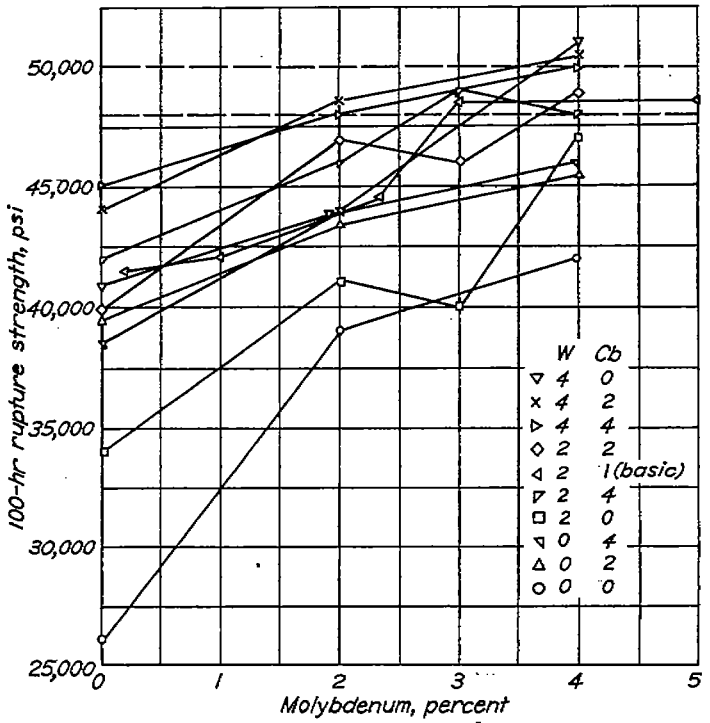


FIGURE 33.—Summary of influence of molybdenum on 100-hour rupture strength at 1200° F of 10 tungsten-columbium modifications. Horizontal dashed lines indicate property range for three basic heats.

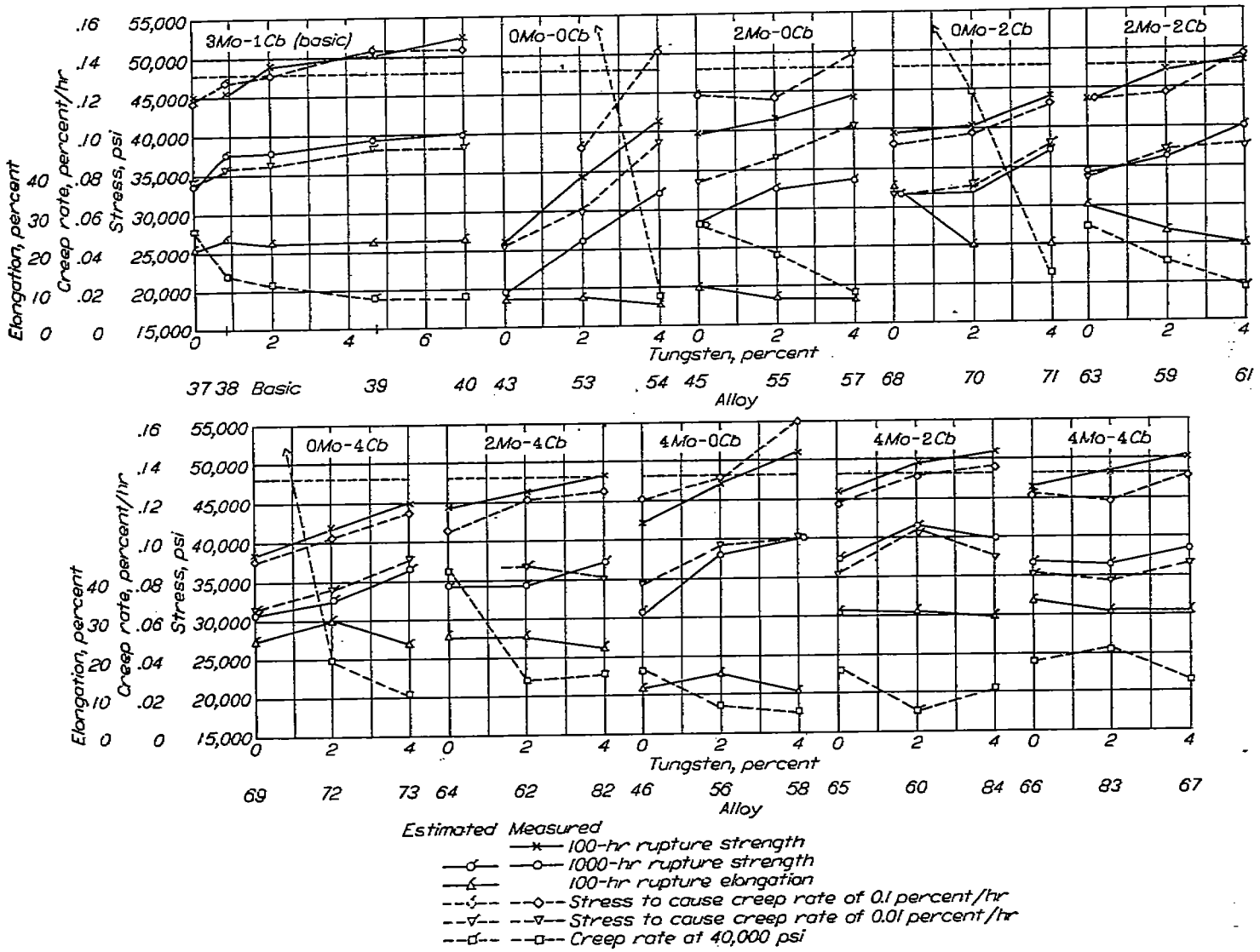


FIGURE 34.—Curves of rupture test data at 1200° F against tungsten content for 10 molybdenum-columbium modifications. Horizontal dashed lines indicate range of 100-hour rupture strength for three basic heats.

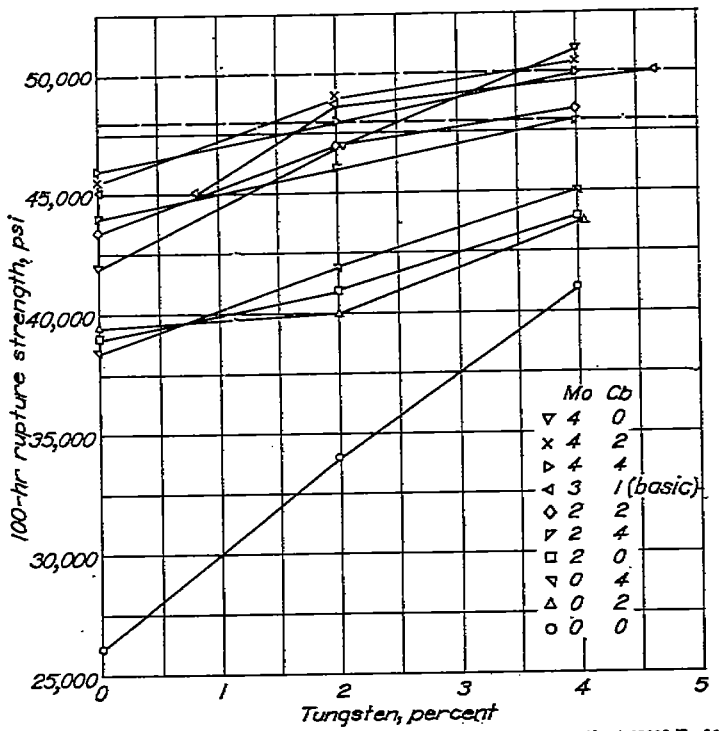


FIGURE 35.—Summary of influence of tungsten on 100-hour rupture strength at 1200° F of 10 molybdenum-columbium modifications. Horizontal dashed lines indicate property range for three basic heats.

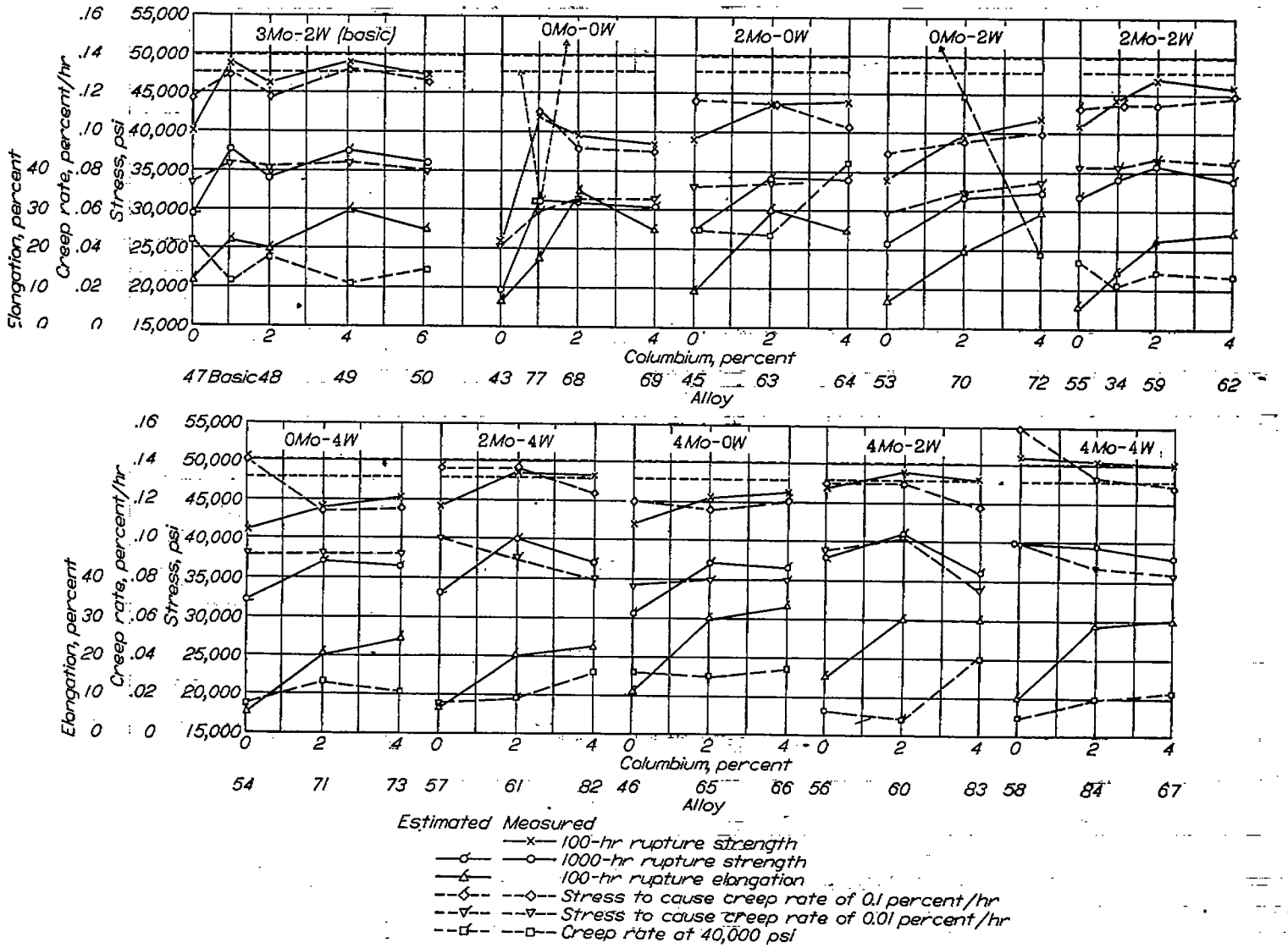


FIGURE 36.—Curves of rupture test data at 1200° F against columbium content for 10 molybdenum-tungsten modifications. Horizontal dashed lines indicate range of 100-hour rupture strength for three basic heats.

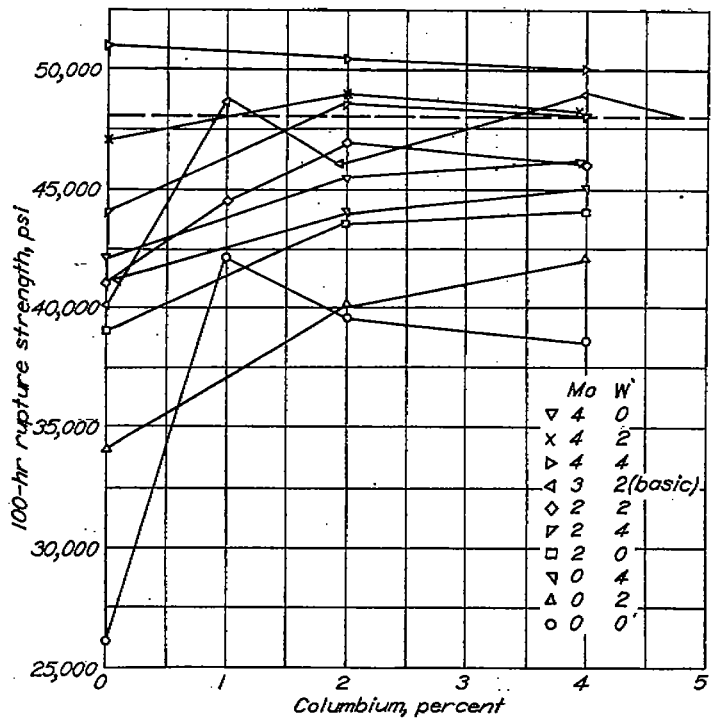


FIGURE 37.—Summary of influence of columbium on 100-hour rupture strength at 1200° F of 10 molybdenum-tungsten modifications. Horizontal dashed lines indicate property range for three basic heats.

Comparative influences of molybdenum, tungsten, and columbium.—Figure 38 shows comparative influences of Mo, W, and Cb on the 100-hour rupture properties at 1200° F for three of the alloy modifications in which these elements were varied simultaneously: 0Mo-0W-0Cb, 2Mo-2W-2Cb, and 4Mo-4W-4Cb. Two of the three elements are constant while the third is varied from 0 to 4 percent giving a family of three curves with one common composition. The 100-hour rupture strengths varied from 26,000 to 51,000 psi. Comparative influences of elements on rupture strengths were as follows:

(1) For the family of curves in which there is a common alloy with a composition of 0Mo-0W-0Cb, a Cb addition of 1 percent gave the highest strength; higher Cb lowered the strength. Increasing Mo and W increased strength continuously with Mo having the greatest influence. The strengths of the alloys on this family of curves were at a lower level, 26,000 to 42,000 psi, than those for the other two alloy modifications.

(2) For the 2Mo-2W-2Cb modifications, having strengths between 40,000 and 49,000 psi, Mo had the greatest strengthening influence; Cb was next, but strength dropped off above 2 percent Cb; and W produced a consistent, but the least, over-all strength increase.

(3) The 4Mo-4W-4Cb modifications were at the highest strength level of the three, 45,000 to 51,000 psi. Additions of Mo and W had about the same strengthening influence while Cb tended to lower strength.

The effects of the elements on 100-hour rupture elongations, also shown in figure 38, were the same for all three levels of composition: W tended to lower elongation slightly; Mo raised the elongation slightly; and Cb additions from 0 to 1 or 2 percent markedly raised the elongation from values below 10-percent elongation to 20 percent or higher.

The combined influence on 100-hour rupture strength of Mo, W, and Cb, on a total weight percent basis is shown in figures 39, 40, and 41. Each of these graphs contains the same points plotted to the same coordinates, these points representing all of the alloys in the testing program in which Mo, W, and Cb were varied, the remainder of the basic analysis being constant. The difference between the graphs is in the method of joining the points. In figure 39 the points are joined to give curves showing the effect of Mo varied from 0 to 4 percent for 10 constant ratios of W and Cb. In figure 40 the points are joined to show the effect of W and in figure 41 the curves show the effect of Cb.

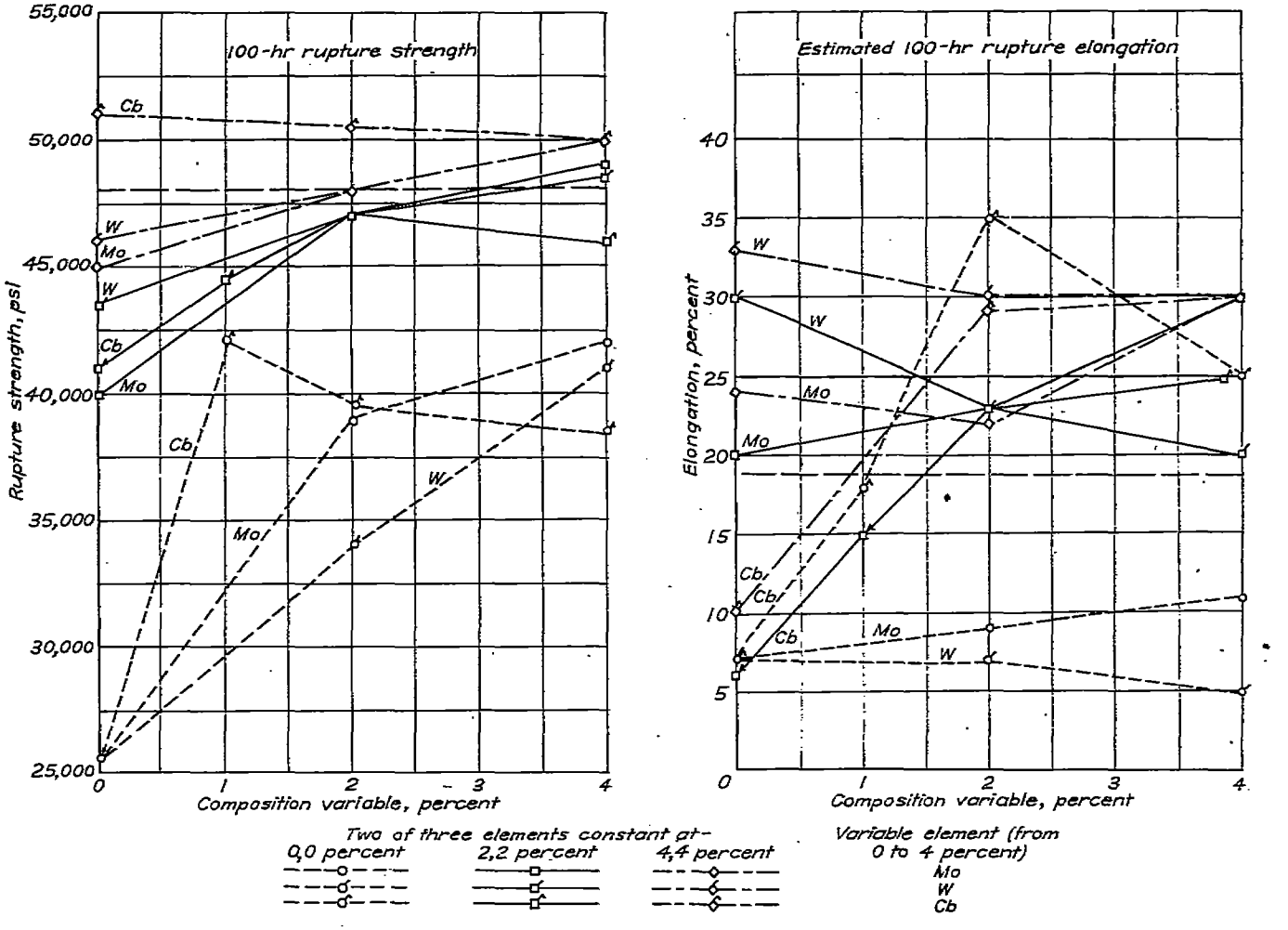


FIGURE 38.—Comparative influences of molybdenum, tungsten, and columbium on 100-hour rupture properties at 1200° F of modified alloys. Horizontal dashed lines indicate property range for three basic heats.

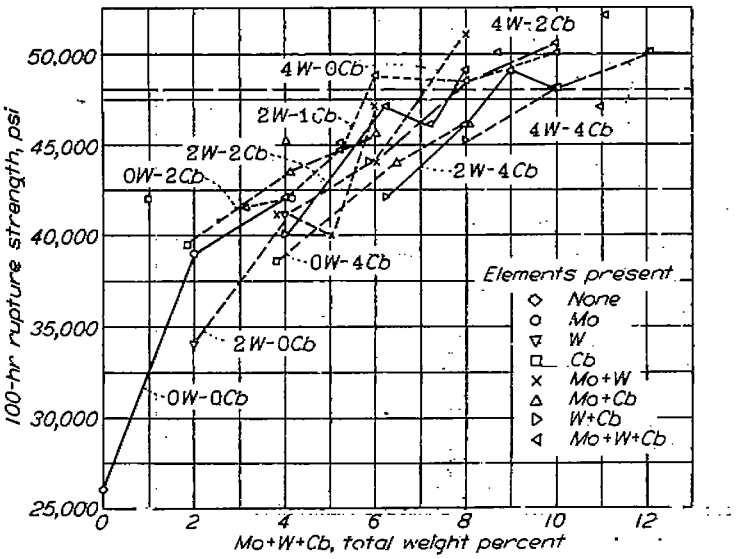


FIGURE 39.—Influence of molybdenum additions (0, 2, and 4 percent) on a total weight percent basis of molybdenum, tungsten, and columbium on 100-hour rupture strength at 1200° F of modified alloys. Horizontal dashed lines indicate property range for three basic heats.

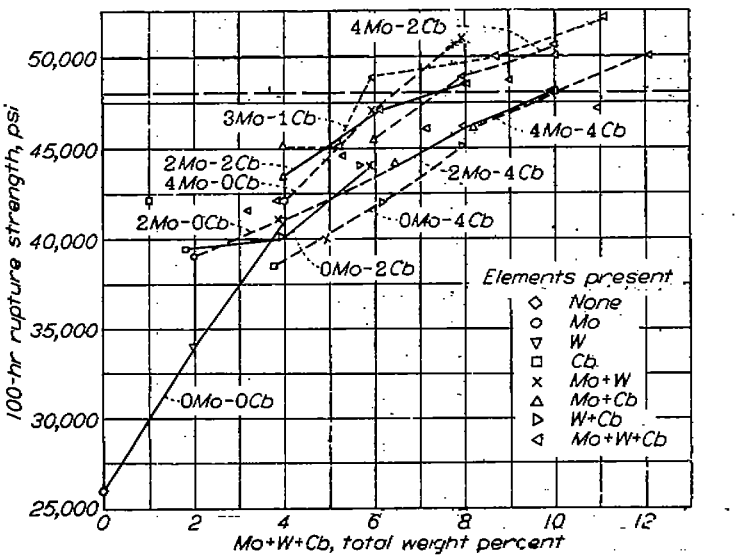


FIGURE 40.—Influence of tungsten additions (0, 2, and 4 percent) on a total weight percent basis of molybdenum, tungsten, and columbium on 100-hour rupture strength at 1200° F of modified alloys. Horizontal dashed lines indicate property range for three basic heats.

The array of points in these graphs indicates the general strength increase resulting from increasing the total alloy content of Mo plus W plus Cb from 0 to 12 percent. The main reason for the scatter of the points is evident from comparison of the curves in the three graphs. Both Mo (fig. 39) and W (fig. 40) produce strength increases over the entire composition range, while the Cb curves (fig. 41) above 1 and 2 percent Cb flatten out and cut horizontally across the property range. This emphasizes the unique influence, shown in previous graphs, of Cb on rupture strength.

The comparative influence on 100-hour rupture strength of Cb in relation to the influence of Mo and W is broken down further in the graphs of figure 42. Curves at constant Cb contents of 0, 1, 2, and 4 percent indicate the combined strengthening influence of Mo plus W. Curves representing this influence have been drawn and are summarized in the bottom graph in which Cb content is the parameter. The short-dashed curves on the upper four graphs represent the influence of Mo and the long-dashed curves, the influence of W at constant amounts of the other element.

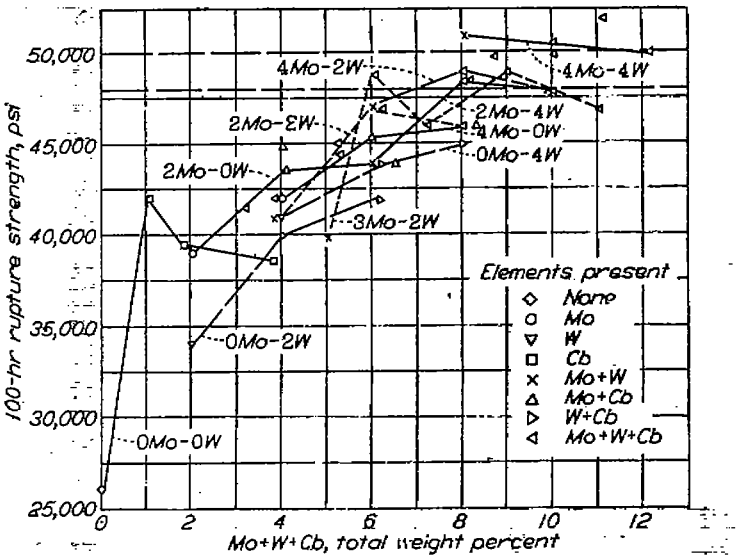
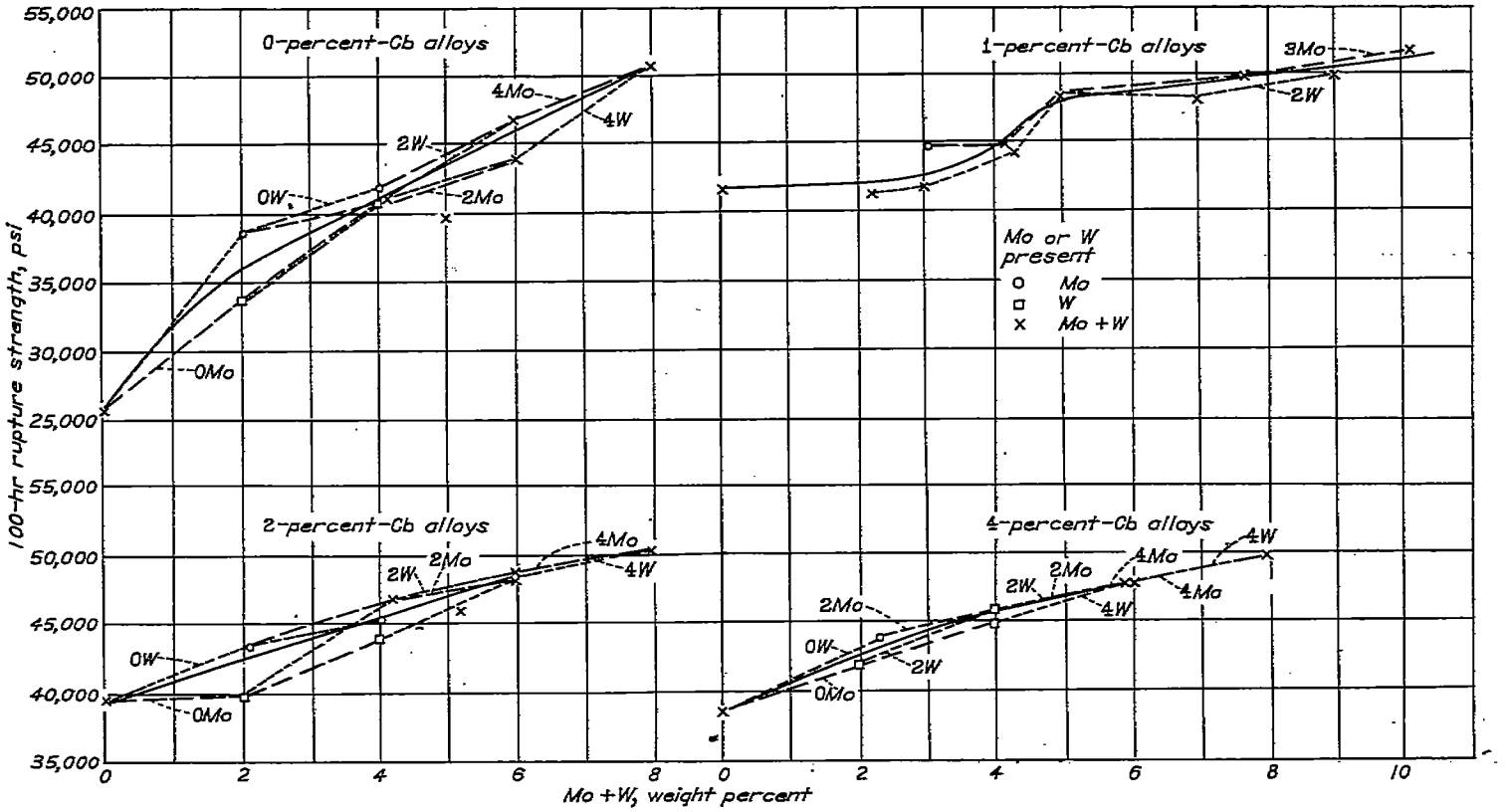


FIGURE 41.—Influence of columbium additions (0, 2, and 4 percent) on a total weight percent basis of molybdenum, tungsten, and columbium on 100-hour rupture strength at 1200° F of modified alloys. Horizontal dashed lines indicate property range for three basic heats.

The spread in data in the graphs of figure 42 was much less than that in figures 39, 40, and 41 in which the influence of Cb was not separated. When Mo is added in lower percentages it has the greater strengthening influence and W has the greater influence at higher percentages. These trends cause the curves representing the Mo and W influences to turn toward the center of the range representing the data spread at the higher percentages of these two elements and thus to strengthen the reliability of the single curves representing the data at constant Cb contents.

The summary curves in figure 42 indicate that additions of more than 1 percent of Cb were of no benefit to the alloys. When approximately 8 percent total of Mo plus W was present any addition of Cb was of no benefit to the strength. As has been noted previously, however, Cb additions of 1 percent markedly improved the rupture ductility over that of the 0-percent-Cb alloys, regardless of the influence of Cb on strength.

From the summary curves in figure 42 it is possible to predict the 100-hour rupture strength of alloys with any combination of Mo and W, at the four Cb levels, within the composition range in which these elements were investigated. Such predictions for alloys falling within the range of 0 to 1 percent Cb are not possible because data were not obtained. Based on the data, the largest error in these predictions would be in the vicinity of 2 percent total of Mo plus W. This error could be as much as 2500 psi, which is not large compared with the 2000-psi range in properties found from heat to heat of the basic alloy. Such predictions of course are tempered by the limitations of the investigation, particularly by the limitation of only one condition of preparation and heat treatment.



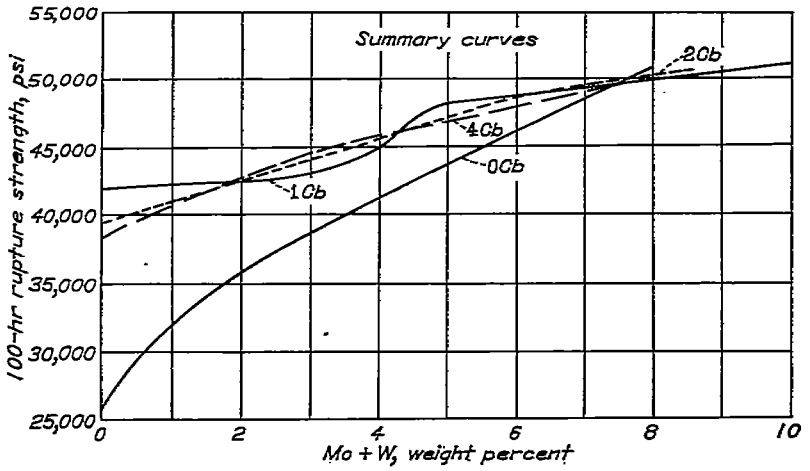


FIGURE 42.—Influence of molybdenum and tungsten additions on a total weight percent basis on 100-hour rupture strength at 1200° F of columbium modifications.

SUMMARY OF INFLUENCE OF CHEMICAL COMPOSITION ON RUPTURE TEST CHARACTERISTICS

Individual variations of elements.—Figure 43, which summarizes the rupture properties at 1200° F for variations of one element at a time in the basic analysis, indicates the following influences of chemical composition for alloys melted, forged, and heat-treated under the conditions of this investigation:

(1) Variation of C and Mn had no appreciable influence over the ranges examined.

(2) Very low N caused somewhat low rupture strength and intermediate N produced strengths slightly above normal.

(3) The effect of Si was unique in that increasing amounts lowered rupture strength and increased elongation.

(4) Increasing amounts of all other elements resulted in increased rupture strength over the complete range of variation except Ni, Co, and Cb which apparently reached a saturation content for rupture strength at 10, 20, and 1 percent, respectively. Additions of Cb caused a pronounced increase in rupture elongation

(5) A saturation point for Mo and W was approached at the 3- and 2-percent levels of the basic alloy although improvements were obtained by further additions of these elements. The element Mo increased and W had no effect on elongation.

(6) The rupture strength was markedly increased by additions of Cr from 10 to 30 percent, and rupture elongation was decreased.

(7) Relative magnitudes of 100-hour rupture-strength improvements were: 20 percent Cr increased strength 13,000 psi; 7 percent Mo, 8500 psi; 7 percent W, 7000 psi; and 1 percent Cb, 8500 psi. The addition of 1 percent of Si decreased strength 5000 psi.

The following modifications had strengths appreciably below that of the basic alloy: 1.2 percent Si, 1.6 percent Si, 10 percent Cr, 0 percent Co, 10 percent Co, 0 percent Mo, 1 percent Mo, 2 percent Mo, 0 percent W, 1 percent W, and 0 percent Cb. The only alloys which had appreciably higher strengths than the basic alloy were those with 30 percent Cr and 7 percent W. From this it appears little was done to improve the 1200° F rupture strength of the basic alloy in the single condition of treatment studied, that rather wide individual variations of the elements can be permitted, particularly to higher values than those in the basic analysis, without appreciably altering properties, and that Cr, Co, Mo, W, and Cb are necessary for high strength.

Simultaneous variations of molybdenum, tungsten, and columbium.—The summarized influences on rupture properties at 1200° F of simultaneous variations of Mo, W, and Cb in the basic analysis, shown in figure 42 for the 100-hour rupture strength, serve to emphasize the general findings when these elements were varied individually. These influences are as follows:

(1) The absence of Mo, W, and Cb yielded an alloy with very low rupture strength, 26,000 psi for rupture in 100 hours.

(2) Separate additions of Mo, W, or Cb in amounts up to 4 percent to the 0Mo–0W–0Cb analysis raised the 100-hour rupture strength up to at least 40,000 psi as compared with 49,000 psi for the basic alloy.

(3) Simultaneous additions of the three elements in amounts up to 4 percent at least doubled the strength of the 0Mo–0W–0Cb analysis. Such additions did not, however, yield alloys with properties which were outstandingly better than those of the basic alloy.

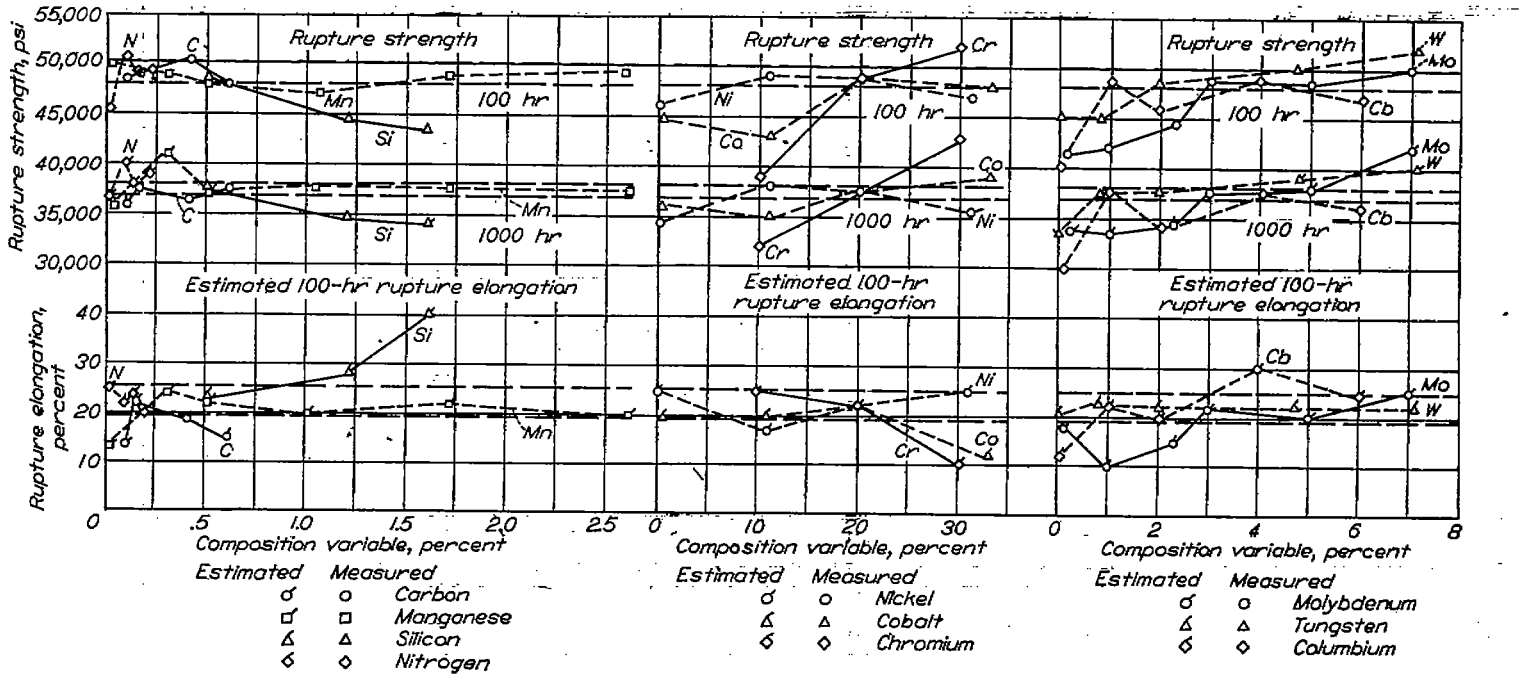


FIGURE 43.—Summary of influence of variation of individual alloying elements in basic alloy on 100-hour and 1000-hour rupture strengths and 100-hour rupture elongation at 1200° F. Horizontal dashed lines indicate property range for three basic heats.

(4) Additions of Mo and W to 4 percent raised strength progressively. Additions of more than 1 percent of Cb, however, were not beneficial to strength.

(5) Additions of Mo and W had little effect on rupture test elongation. Alloys containing no Cb had consistently low elongation and additions of 1 to 2 percent Cb markedly increased the elongation.

Relation of rupture and creep properties.—It was noted in table X and in the graphs of properties against composition that the stresses causing a minimum creep rate of 0.1 percent per hour were similar in magnitude to the 100-hour rupture strength. In general it was also noted that changes in rupture strength were accompanied by similar changes in creep properties except for alloys with relatively low rupture elongation, particularly alloys not containing Cb, where total elongation appeared to have relatively greater influence on fracture time.

It appears, therefore, that the rupture strengths of the alloys were controlled by their inherent resistance to creep, as measured by the stresses based on minimum creep rates obtained during the rupture test and their total elongation to fracture.

DISCUSSION OF RESULTS

The results indicate that by careful control of processing conditions the high-temperature characteristics of forged Cr–Ni–Co–Fe–Mo–W–Cb alloys can be related to systematic variations of chemical composition and that major changes in rupture characteristics at 1200° F accompany certain variations in composition. A summary of the influence of alloy modification on the 100-hour rupture strength at 1200° F is shown in figure 44 for the elements (Si, Cr, Ni, Co, Mo, W, and Cb) producing significant changes. There were also marked changes with alloy modification in such other metallurgical properties as microstructure, hardness, and melting and forging characteristics which indicate reasons for the observed influence of composition on rupture characteristics.

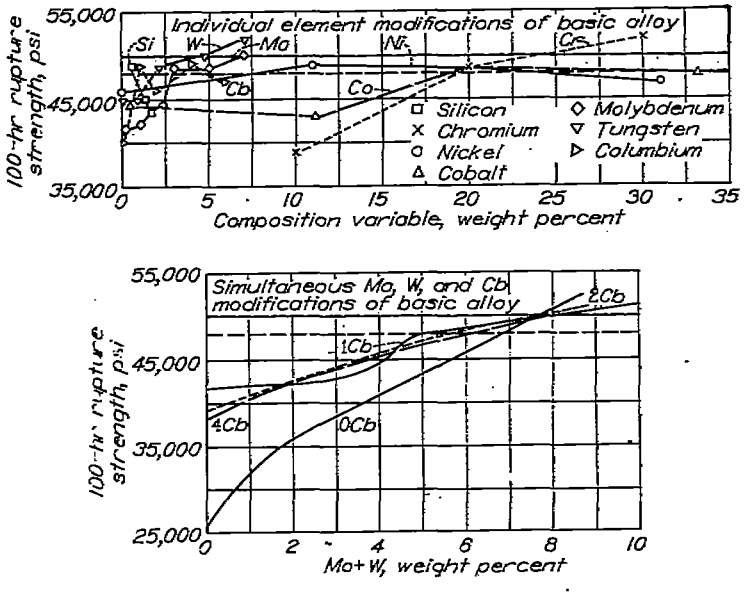


FIGURE 44.—Summary of influences of alloy modification on 100-hour rupture strength at 1200° F. Horizontal dashed lines indicate property range for three basic heats.

The range in 100-hour rupture properties associated with the composition variables studied was 26,000 to 52,000 psi for the rupture strength and 5 to 40 percent for the rupture elongation. These ranges compare with properties of other heat-resisting alloys as shown in table XI. It is seen that the rupture properties of the basic alloy can be varied over just as large a strength range, but at a higher level, as a result of variations in prior processing as was obtained by composition modifications for a single processing condition. The strength of the modification (0Mo–0W–0Cb) at the low end of the composition strength range is similar in magnitude to strengths of the standard heat-resisting alloys of the 18Cr–8Ni type. Wide property ranges are also shown for seven superalloys studied in a research program on a large number of alloys in which it was found that no accurate comparision could be made between alloys on the basis of chemical composition because prior processing conditions were not controlled. The present investigation has shown that certain variables in melting and hot-working procedures definitely result in major variations in high-temperature properties and that these variations probably cannot be completely removed by subsequent heat treatment.

LIMITATIONS OF DATA

Interpretation of the results can be made only subject to the limitations initially placed on the investigation and those which developed as a result of the investigation. The major limitations which appear to be of significance are listed below:

(1) A limited number of composition variables were studied. Simultaneous variations of elements were made only for Mo, W, and Cb. While certain of the composition variables studied showed significant influences on properties there was only limited indication of what to expect from simultaneous variation of other combinations of the elements in the alloy.

(2) Comparisons between alloys were limited to properties in one condition of prior processing. It was shown that the control exercised over processing was sufficient to obtain reliable reproducibility of properties between heats of the basic alloy. But it was also shown that, unless all the processing procedures, including both melting and hot-working conditions, are controlled, variability in properties may result. Reasons for this variability were not evident from this investigation. The relative influences of processing on alloys other than the basic are not known. Because only one processing condition and heat treatment were used for all the alloys no indication could be obtained of the optimum properties which would be expected for the alloys by variation of treatment.

(3) Limitations are also imposed in the interpretation of the influence of composition on high-temperature characteristics in general by the fact that only one type of test was used to evaluate properties and this at only one temperature. Testing time was also usually limited to that necessary to establish the 100-hour rupture strength.

In the discussion of the results of this investigation cognizance is made of these limitations.

COMPARATIVE RUPTURE AND CREEP CHARACTERISTICS

The main reason for consideration of the creep data from time-elongation curves of the rupture tests was to determine if creep resistance or elongation best correlated with the rupture strengths. It was observed that in general the stresses to cause a creep rate of 0.1 percent per hour were of the same order of magnitude and followed the same trends with composition variation as the 100-hour rupture strengths. The main exception to this observation was in the alloys in which Cb was systematically varied from 0 to 4 percent in which case the stresses for constant creep rates remained constant or decreased with increasing rupture strength (see fig. 36). It seemed apparent, therefore, that the marked influence of Cb additions from 0 to 1 or 2 percent on increasing inherent ductility was responsible for the unusual relation between creep and rupture properties. While stresses at a constant creep rate of 0.1 percent per hour were of the same order of magnitude as the 100-hour rupture strength, these stresses were higher for the 0-percent-Cb alloys (low elongation) and equal to or less than the rupture strength for the 1- and 2-percent-Cb alloys (high elongation). Apparently, therefore, the influence of Cb on changing the rupture strength was less dependent on the resistance to creep, as measured by stress at constant creep rate, than on the relative ability of the alloys to deform before fracture.

Figure 45 shows, for all the alloys studied, the variation of the 100-hour rupture strength with stress to cause a creep rate of 0.1 percent per hour. Average curves are drawn representing the points indicating the low (5 to 10 percent), intermediate (11 to 19 percent), and high (20 to 40 percent) 100-hour rupture-elongation levels encountered. While there are ranges for the rupture-creep property relations it is noted that the range representing high elongations is definitely at a higher level than that representing low elongations. The range for intermediate elongations overlaps both those for high and low elongations. These ranges, while partially the result of variation in actual elongation within the range, particularly at the lower elongations, are believed to be mainly caused by the limitation of the reproducibility of properties from heat to heat of a given analysis and by the fact that the creep properties in many cases were estimated from only a small amount of time-elongation data. Another possible cause of the ranges could be that for certain alloys the stress at constant creep rate is not an accurate representation of the creep resistance.

In general it appears, however, that the stress to cause a creep rate of 0.1 percent per hour is a measure of creep resistance which does control the 100-hour rupture strength, that the relation is linear and represented by a line with a slope of approximately 45°, but that the level of rupture properties at a given creep resistance is dependent upon inherent ability to deform before fracture. Specific examples of the relative dependence of rupture strength on stress at constant creep rate are as follows (see fig. 45):

(1) At a constant 0.1-percent-per-hour creep strength of 45,000 psi (which means all alloys on this vertical 45,000-psi line have constant creep resistance) alloys having high elongation will take longer to fracture than those having low elongation. The 100-hour rupture strength will be approximately 5000 psi higher for an alloy with 20-percent or more total elongation than for an alloy with 5- to 10-percent elongation.

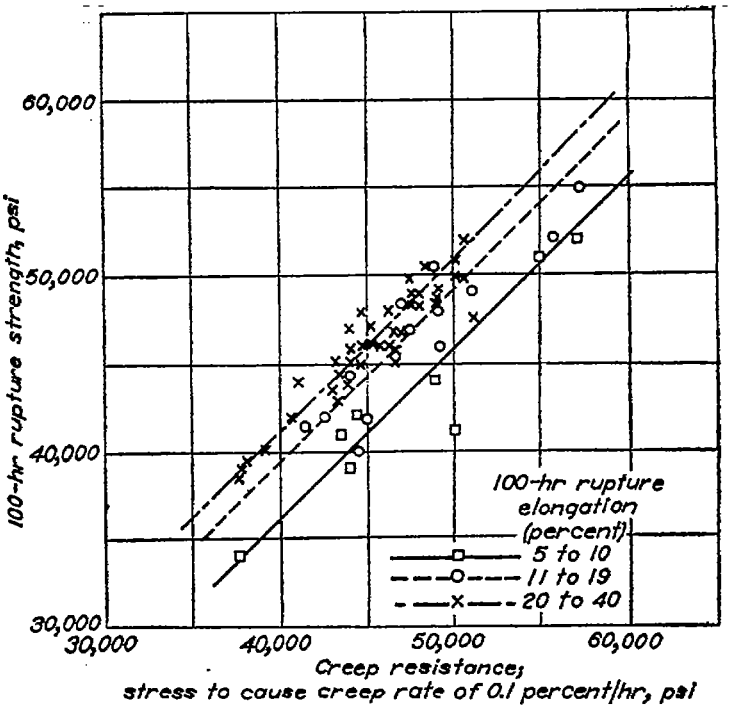


FIGURE 45.—Influence of creep resistance and 100-hour rupture elongation on 100-hour rupture strength at 1200° F of modified alloys.

(2) The 100-hour rupture strength must remain the same, for example at 45,000 psi, even though the stress at constant creep rate, or creep resistance, decreases by approximately 5000 psi because the ability to deform prior to fracture increases at the same time.

It is noted in figure 45 that the level of the relation between rupture strength and creep resistance is dependent on total elongation only at the lower elongations. The reason for this is the difference between the time-elongation curves for ductile and brittle materials. For ductile materials toward the end of the period of increasing creep rate (third-stage creep) there is a large amount of deformation in a very short time period. In this case the total elongation has a relatively small time dependence, the elongation having little influence on the rupture time. For brittle materials, on the other hand, the elongation change with time is more gradual near the end of the test. Thus with the same creep resistance the rupture time is more dependent on the relative inherent ability of the materials to deform before fracture.

The points in figure 45 that indicate elongations between 5 and 10 percent represent the alloys which did not contain Cb. One point at the highest stress represents the high-Cr alloy which had low ductility. It is evident that the unusual relation between creep and rupture properties observed for alloys in which Cb was varied systematically was the result of the added influence of total elongation on rupture strength.

It can be concluded that the rupture-strength variations with chemical composition observed in this investigation were the result of the changes in inherent creep resistance caused by alloy additions except for composition variations for which total elongation to fracture changed from low (5 to 10 percent) to high (above 20 percent) in which case the greater ability to deform before fracture also improved the rupture strength.

INTERPRETATION OF INFLUENCE OF CHEMICAL-COMPOSITION VARIABLES ON RUPTURE PROPERTIES AT 1200° F OF MODIFIED ALLOYS

Interpretation of the manner in which the composition variations influence the high-temperature properties of these alloys by controlling the inherent creep resistance and the ability to deform before fracture can be made in view of simultaneous effects noted in other metallurgical characteristics. The following observations of such effects were made:

(1) Only the following nine alloys of the 63 different compositions studied developed a pronounced amount of visible microstructural precipitate during aging:

Alloy	Composition variable (percent)
15	0.40C
16	0.60C
52	30Cr
29	0Co
49	3Mo–2W–4Cb
50	3Mo–2W–6Cb
82	2Mo–4W–4Cb
83	4Mo–2W–4Cb
67	4Mo–4W–4Cb

Except for the 30-percent-Cr alloy, the additional precipitate, resulting from either increased C or Cb or decreased Co, did not have any apparent beneficial effect on strength. The matrix precipitate which occurred during aging of the 30-percent-Cr alloy was of a different type from that of other alloys with heavy precipitation, being preferred rather than random. The hardness increase during aging of the 30-percent-Cr alloy was exceptionally large. Thus the substantial strength increase with the addition of Cr from 20 to 30 percent appeared to result, at least in part, from the aging characteristics caused by higher Cr.

(2) Neither Mo nor W, when varied from 0 to 7 percent in the basic alloy containing 1 percent Cb, showed the least effect upon the relative amounts of visible precipitate occurring during aging. However, accompanying the increases in these elements were improvements in creep resistance and rupture strength over the entire composition range. Likewise Mo and W, when varied simultaneously from 0 to 4 percent, produced no increase in amount of visible aging precipitate in alloys containing 2 percent or less of Cb. Creep-resistance and rupture-strength improvements were considerable and continuous in the 20 series of alloys in which either Mo or W was increased systematically. In 16 series of alloys, there was no appreciable change in the effect of these elements on aging precipitate present. The remaining four series were those containing 4 percent Cb, and in these the presence of at least 6 percent total of Mo plus W was necessary to increase the amount of visible aging precipitate. The elements Mo and W were similar in effect on properties in these systems as in the systems containing lower Cb. However, these high-Cb alloys did not develop an appreciable relative increase in hardness during aging and had a random rather than the preferred matrix precipitate of the high-Cr alloy which apparently derived its superior strength from the aging reaction.

(3) The Cb produced its major improvement in rupture strength in additions of 1 percent to the 0-percent-Cb alloys. This increase in rupture strength appeared to be the result of the higher ductility caused by Cb rather than an improvement in creep resistance, which Cb additions either did not change or reduced. Additions of more than 2 percent Cb did not add anything to rupture strength because creep resistance was not increased and the rupture elongations were all at a high level where changes in total elongation did not appreciably affect fracture time. Microstructural examinations indicated that Cb, or the compound it caused to form, was at most only partially soluble at temperatures up to 2200° F. Limited solubility appeared to be the reason that there was no improvement in creep resistance when Cb was added. The major changes in microstructure caused by Cb—increasing the amount of insoluble constituent, changing the mode of aging precipitation from preferred to random, and refining the grain size—appeared to be associated with the marked increase in rupture elongation resulting from Cb additions. It is noted that low rupture elongation was also associated with the preferred type of aging precipitate in the 30-percent-Cr alloy. Rupture-strength improvements with Cr additions, however, were the result of increased creep resistance which was not the case for the Cb-modified alloys.

Additions of 4 percent or more of Cb in the presence of 6 percent or more of Mo plus W caused a marked increase in the amount of random matrix precipitation during aging. This additional precipitate added nothing to strength and did not appreciably increase the relative hardening during aging. However, the strength increases accompanying additions of Mo and W in this region continued to be consistent with those which appeared to be the result of solid-solution effects for the lower-Cb alloys.

(4) There were certain similarities between the effects of C and Cb on the amount of excess constituents present in the microstructures. While the excess constituents resulting from C and Cb additions were not necessarily the same phases, the appearance of large amounts of excess constituents, both in the solution-treated condition and during aging of the higher-C modifications, had little effect on properties. Apparently C combines with elements normally present in the solid solution to form certain of the observed excess constituents. In view of this the inherent matrix strength of the material should be reduced. This effect appeared to be the case for the 0.60-percent-C alloy which did show a tendency toward lower strength. However, balancing factors, which could hold strength up to a certain extent, could be that some strengthening resulted from the very heavy aging precipitation, or that C did not combine with the major elements causing the strength.

(5) Additions of Co from 0 to 20 percent which tended to raise creep resistance, and thus rupture strength of the alloy, decreased the amount of visible aging precipitate, apparently increasing the solubility of the strengthening elements. Further addition to 32 percent Co increased creep resistance but did not appreciably improve rupture strength because of the lower rupture elongation.

(6) The major strengthening effect of Ni was between 0 and 10 percent. The addition of 10 percent Ni changed the alloy from an unbalanced ferrite-austenite composition of the 0-percent-Ni alloy to the austenitic-matrix-type alloy typical of all the other alloys studied. By preventing the formation of the weak ferrite-sigma-type phase 10 percent Ni caused the elements in this phase to enter the solid solution of the matrix, thus increasing the creep resistance and rupture strength.

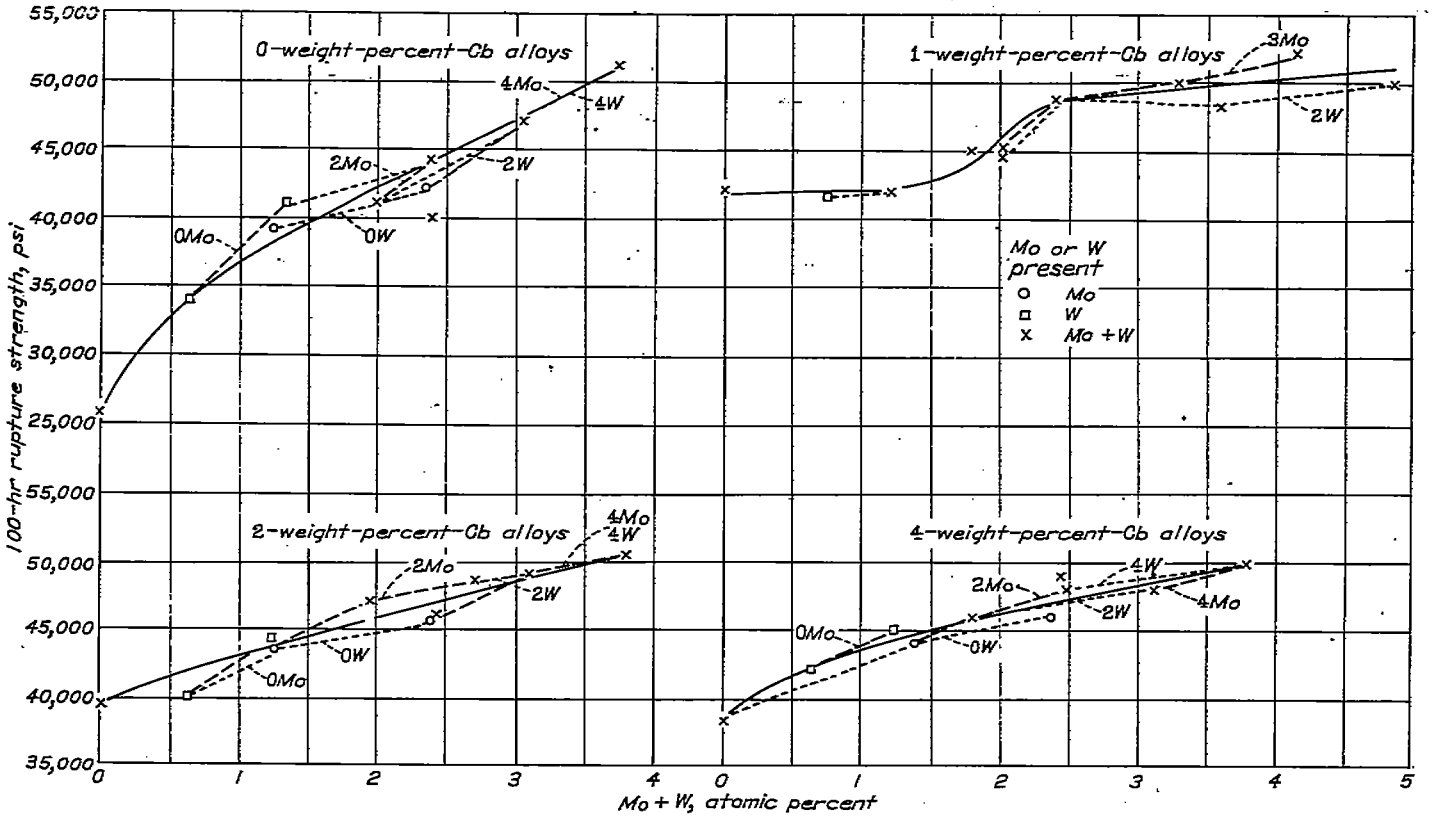
Each of the above observations of relations of metallurgical characteristics and high-temperature properties points to the conclusion that the major strengthening effects produced by alloy variations in this investigation were the result of the elements entering into solid solution in the alloys. The two outstanding examples of solid-solution strengthening were for Mo and W. These elements would enter the solid solution by substitution, as also would Cr, Ni, and Co, for atoms of Fe, which they replaced in the alloys. The atoms of Cr, Ni, and Co are all about the same size as Fe atoms and could substitutionally enter the crystal lattice of the solid solution with ease. Substitution of the larger or incongruous atoms of Mo and W into the lattice, however, would necessarily set up a strained condition. Such strains in the lattice would interfere with the flow conditions during creep, thus giving the material higher creep resistance. The role of Cb, which also has relatively large atoms, is complicated by the high affinity of Cb for C and by the apparent low limit of solubility of Cb, or the phase which it forms, in the basic alloy system studied. The substitution of the similarly sized elements, Cr, Ni, or Co, into the lattice would not set up particularly strained conditions and thus strengthening from solid-solution effects would not result. This has also been demonstrated by a recent investigation of Fe-Cr-Ni alloys (reference 2). The effects of Ni and Co on properties and microstructure appeared to be the result of these elements influencing the matrix solubility of other elements, the two most probable other elements being Mo and W. As noted, Cr apparently increased strength, at least in part, by aging effects.

It would appear therefore that as the number of incongruous atoms entering solid solution increased the strength should increase proportionately. The composition modifications studied most thoroughly in this investigation were those involving the simultaneous variation of Mo, W, and Cb. The modifications in which Mo and W additions caused strength increases were pointed out as examples of strengthening as a result of solution effects. Figure 46 shows the influence on the 100-hour rupture strength of total atomic additions of Mo and W to alloys containing four levels of Cb. This figure is similar to the presentation in figure 42 of the same data on a weight percent basis. The solid-line curves drawn represent the data even better, however, when presented on an atomic percent basis. Trends in the summary curves, with Cb content as a parameter, are the same in both presentations, Cb above 1 percent being of no benefit to strength. In general, the curves in figure 46 show a continuous and approximately proportionate increase in strength with atomic additions of Mo and W. This is consistent with what would be expected if solid-solution effects are responsible for the strength increase.

The points in the graphs of figure 46 have been connected so as to show the comparative effects of Mo and W, the short-dashed lines showing the Mo effects and the long-dashed lines the W effects. There is a general trend indicated by these curves for W to cause a relatively greater strengthening than does Mo. This means that, although the average solid-line curves drawn do represent quite well the data in the graphs for the composition ranges studied, additions of atoms of W to the solid solution had a greater strengthening effect than equivalent atomic additions of Mo.

Exceptions to the solid-solution strengthening effect, particularly that of the 30-percent-Cr alloy, were noted previously. One other major exception, which does not agree with the conclusion that strengthening resulted from solid-solution effects, was that encountered in the influence of Si variations. The effect of Si, varied from 0.5 to 1.6 percent, was to lower strength properties markedly and to increase total deformation to fracture. There was no apparent change in microstructure produced by Si. It was also observed that the high-Si alloy was the only alloy of all those studied which did not show any increase in hardness during aging.

A clue to the apparently anomalous effect of Si is to be found in the results of the effect of deoxidation practice during melting on the properties of the basic alloy. Table VI shows a 100-hour rupture strength of 52,000 psi for alloy 74 which was not deoxidized before pouring. This strength of 52,000 psi is to be compared with the strength range of 48,000 to 50,000 psi for the basic alloy deoxidized in the normal manner with calcium-silicon deoxidant and with the 55,000-psi rupture strength of alloy 75 which was deoxidized with a zirconium-silicon-iron deoxidant. These results indicate that a marked influence may occur on properties of a given alloy as a result of variation of melting procedure. The element Si definitely plays an important role in melting, particularly in deoxidation practice. One heat (alloy 27) in which the charge was aimed at yielding 0.25 percent Si in the basic analysis and which was deoxidized in the normal way with 15 grams of calcium-silicon alloy resulted in 0.58 percent Si and gave properties typical of the basic alloy. Another heat (alloy 79) aimed at 0.25 percent Si, but in which only 5 grams of calcium-silicon deoxidant were used, resulted in a blow hole at the ingot center. It thus appears that inherent characteristics, imparted to the material during melting, not removed by subsequent processing, and not evident in microstructure, could be responsible for the unusually low strengths of the high-Si alloys.



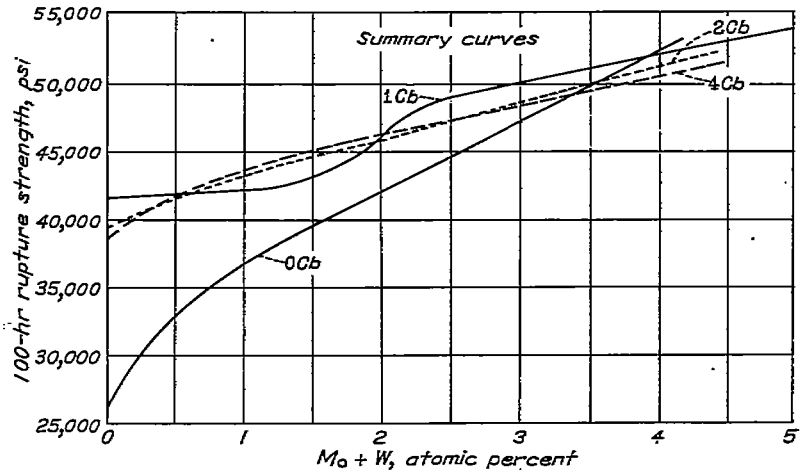


FIGURE 46.—Influence of molybdenum and tungsten additions on a total atomic percent basis on 100-hour rupture strength at 1200° F of columbium modifications.

The basic alloy has been shown by previous investigations to be an alloy which does not develop appreciable strengthening at high temperatures as a result of visible precipitation reactions during aging after solution treatment (references 1 and 3). Evidence presented in this discussion indicates that the major strengthening effects accounting for the good strength of the basic alloy result from additions of incongruous atoms of elements to the solid-solution lattice of the austenite-type matrix.

Another characteristic evaluated for the alloys was the relative forgeability. Although this evaluation was only qualitative the indications were that high-temperature characteristics as measured by rupture tests at 1200° F were in general reflected at temperatures of 1800° to 2200° F in the relative forgeability of the alloys. Alloys which had higher rupture strengths as a result of greater resistance to creep also tended to be less plastic in the forging range. The main exception to this was for Mo additions which apparently did not affect forgeability. The alloys not containing Cb, which had quite low rupture elongation, tended to crack during forging.

OPTIMUM ALLOY CONTENT AND COMPOSITION RANGES

In addition to the main objective of determining the influence of chemical-composition variations on rupture properties at 1200° F, the present investigation was also intended as an initial step toward establishment of optimum compositions for alloys of the basic-alloy type. Interpretation of the results for this purpose, however, is subject to the limitations of the number of compositions studied and the single preparation condition placed on the investigation.

While the over-all property range obtained was quite wide and information was obtained concerning the influence of each element on properties, no alloy was obtained which had properties which were outstanding compared with those of the basic analysis.

It was shown that most of the elements present in this analysis are necessary to yield the best properties, but that variations over relatively wide ranges can be made of one element at a time in the basic analysis without appreciably altering properties. Indications are that Ni can be varied from 10 to 20 percent, C from 0.08 to 0.60 percent, Co from 20 to 32 percent, Cb from 1 to 4 percent, Mn from 0 to 2.5 percent, and N from 0.08 to 0.18 percent in the basic analysis without appreciably affecting properties. It was also shown that Cr, Mo, and W are necessary for maintenance of properties and that Cb in additions not beyond 1 percent is necessary for high ductility characteristics. It is possible, however, to obtain strengths as high or higher than that of the basic alloy without the use of Cb but this involves increasing Mo and W.

Seemingly the only major reduction in required alloy content in the basic alloy, which could be made without appreciably lowering properties, is that Ni could be lowered to 10 percent. Possibly Cb could be decreased a few tenths of a percent below 1.

NATURE OF PHASES PRESENT IN EXPERIMENTAL ALLOYS

As yet little is known concerning the nature of the phases present in alloys of the type investigated. It is generally accepted that the austenite-type matrix is a solid solution which is saturated with respect to certain unidentified constituents at both the solution and aging temperatures. It is also known that Cb, Cr, Mo, and W are prone to form carbides or nitrides under certain conditions and that intermetallic compounds, such as the Fe-Cr sigma phase, and also a ferrite-type phase, are possibilities of microconstituents.

There was a limited amount of evidence gained from the microstructural studies of this investigation concerning the nature of the phases encountered. There is evidence that C entered into the reaction forming the phase which was at least partially insoluble at the solution-treatment temperature and that Cb was the other significant element in the formation of this phase. It also appeared that C was an important constituent of the precipitate which occurred during aging.

Evidently Cr contributed to the formation of the phase which precipitated during aging of the 0Mo-0W-0Cb alloy. It appeared that Cr was also quite important in the aging reaction in the 0-percent-Cb alloys since additions of Mo and W to these alloys did not change the appearance or appreciably affect the amount of aging precipitate.

Additions of Cb from 2 to 4 percent in alloys containing high Mo plus W appreciably increased the amount of aging precipitate. Evidently this occurred as a result of exceeding the solubility limit of the solution which caused the rejection during aging of the excess phase from supersaturated solution. The constituents of this phase were not evident from the data, although there was some indication that Mo and W, which apparently increased strength by substitutional entrance into solid solution, probably were not present in the precipitate. The two examples of this were:

(1) Additions of C to the basic alloy increased aging precipitate but did not appreciably lower strength which would be expected if Mo or W were forced out of solution.

(2) Additions of Cb from 2 to 4 percent in alloys also containing at least 6 percent total of Mo plus W did not affect strength appreciably while increasing the amount of aging precipitate. On the other hand, when Mo or W was raised from 2 to 4 percent in alloys containing 4 percent Cb, producing alloys with more aging precipitate, the strength was improved, indicating that Mo and W went into solution.

In the event Mo and W did maintain strength by remaining in or entering into solid solution, it is probable that Cr or some of the other elements with atoms of similar size to Cr were forced out of solution as carbides or intermetallic compounds.

On the basis of the composition range studied for N, it did not appear that this element appreciably affected the microconstituents.

CONCLUSIONS

By the use of careful control over processing conditions this investigation has shown that for forged alloys containing chromium, nickel, cobalt, iron, molybdenum, tungsten, and columbium it is possible to correlate the stress-rupture properties at 1200° F with systematic variations in chemical composition and that a wide range in properties can be obtained by such variations. However, no alloy was obtained which had properties which were outstanding compared with those of the basic analysis.

Subject to the limitations placed on this investigation of the limited number of composition variables studied, the use of only one condition of processing, the evaluation of high-temperature characteristics by only relatively short-time rupture tests at 1200° F, and the use of only microstructural and hardness data to provide interpretation of results, the findings lead to the following conclusions:

1. Carbon (varied from 0. 08 to 0. 60 percent), manganese (0 to 2.5 percent), nitrogen (0.08 to 0.18 percent), nickel (10 to 20 percent), cobalt (20 to 32 percent), and columbium (2 to 4 percent) do not appreciably influence 1200° F rupture properties. Nitrogen (0.004 to 0.08 percent), chromium (10 to 30 percent), nickel (0 to 10 percent), cobalt (0 to 20 percent), molybdenum (0 to 4 percent), tungsten (0 to 4 percent), and columbium (0 to 1 percent) improve the rupture strength. Silicon (0.5 to 1.6 percent) and nickel (20 to 30 percent) lower rupture strength.

2. The rupture-strength variations with chemical composition observed in this investigation were the result of changes in inherent creep resistance, caused by alloy additions, except for composition variations for which total elongation to fracture changed from low (5 to 10 percent) to high (above 20 percent), in which case the greater ability to deform before fracture also improved the rupture strength.

3. The rupture-strength improvements accompanying the increased creep resistance with additions of molybdenum and tungsten apparently are the result of the strengthening influence of these incongruous atoms entering substitutionally into the matrix solid solution. Increased creep resistance with nickel and cobalt additions apparently results from the manner in which these elements improve the solubility of molybdenum and tungsten. The increased creep resistance produced by chromium additions apparently results, at least, in part, from an aging reaction. The creep resistance is not influenced by columbium which enters solid solution to only a very limited extent. Improvements in rupture strength with small additions of columbium result from the greater ability to deform before fracture of alloys containing columbium. The detrimental effect of silicon on strength properties is possibly connected with melting phenomena which are not yet understood.

4. Columbium, chromium, and carbon produce major changes in microstructure. There is limited evidence to indicate that these are the major elements in the present excess constituents in the structure of the alloys.

5. Melting and hot-working conditions have an important influence on the inherent high-temperature properties of alloys of the type studied.

UNIVERSITY OF MICHIGAN,
ANN ARBOR, MICH., *October 26, 1949.*

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TABLE I.—CHEMICAL COMPOSITION OF EXPERIMENTAL ALLOYS

Alloy	Aim modification from basic alloy (percent)			Chemical composition (percent)[a]									
				C	Mn	Si	Cr	Ni	Co	Mo	W	Cb	N
	Basic analysis			0.15	1.7	0.5	20	20	20	3	2	1	0.12
1[b]	Basic			.25	1.54	.40	18.45	19.45	20.35	3.06	2.30	1.05	.11
2[b]	Basic			.24	1.62	.53	18.88	17.20	19.74	2.95	2.26	1.11	.12
3[b]	Basic			.25	1.68	.60	18.99	18.00	20.64	2.77	2.62	1.12	.15
4[b]	Basic			.30	1.87	.74	19.37	19.04	17.95	2.89	2.34	.96	.12
5[b]	0.4 C			.55	1.58	.67	17.53	18.24	20.54	2.63	1.75	1.00	.12
6[b]	0.4 C			.53	1.78	.84	16.27	18.77	18.04	3.32	2.50	.82	.10
7[c]	Basic			0.16	1.40	0.60	20.64	17.34	20.52	3.01	2.34	1.11	0.15
8[c]	Basic			.14	1.46	.41	21.17	20.16	19.81	3.03	2.08	.96	.14
10[c]	Basic			.15	1.40	.52	20.89	20.04	19.30	3.05	1.91	1.03	.17
11[c]	Basic			.16	1.63	.77	21.00	17.13	22.46	3.04	1.97	1.05	.14
12[c]	Basic			.16	1.61	.67	21.04	18.39	22.00	3.03	2.01	1.15	.14
27[d]	Basic			.15	-------	.58	-------	-------	-------	-------	-------	-------	-------
74[e]	Basic			-------	-------	-------	-------	-------	-------	-------	-------	-------	-------
75[e]	Basic			-------	-------	-------	-------	-------	-------	-------	-------	-------	-------
13	0.07 C			0.08	1.80	0.37	20.03	20.84	19.42	3.12	2.10	1.09	-------
14	0.40 C			.36	-------	-------	-------	-------	-------	-------	-------	-------	-------
15	0.40 C			.40	-------	-------	-------	-------	-------	-------	-------	-------	-------
16	0.60 C			.57	-------	-------	-------	-------	-------	-------	-------	-------	-------
17	0.60 C			.60	1.83	-------	18.59	20.25	18.92	-------	-------	-------	-------
24	0.03 Mn			0.14	0.03	-------	-------	-------	-------	-------	-------	-------	-------
19	0.25 Mn			.15	.30	0.38	20.07	20.70	19.82	2.99	2.02	1.13	-------
20	0.50 Mn			.14	.50	-------	-------	-------	-------	-------	-------	-------	-------
21	1.00 Mn			.12	1.04	-------	-------	-------	-------	-------	-------	-------	-------
22	2.50 Mn			.14	2.58	-------	-------	-------	-------	-------	-------	-------	-------
28	1.0 Si			0.13	-------	1.19	-------	-------	-------	-------	-------	-------	-------
80	1.5 Si			.14	-------	1.56	-------	-------	-------	-------	-------	-------	-------
51	10 Cr			0.14	-------	-------	10.18	-------	-------	-------	-------	-------	-------
52	30 Cr			.15	-------	-------	30.51	-------	-------	-------	-------	-------	-------
23	0 Ni			0.15	-------	-------	-------	0.01	-------	-------	-------	-------	-------
25	10 Ni			.16	-------	-------	-------	10.70	-------	-------	-------	-------	-------
26	30 Ni			.14	-------	-------	-------	30.64	-------	-------	-------	-------	-------
29	0 Co			0.19	-------	-------	-------	-------	0.31	-------	-------	-------	-------
30	10 Co			.14	-------	-------	-------	-------	11.09	-------	-------	-------	-------
31	30 Co			.14	1.76	0.79	20.50	20.18	32.60	3.41	2.05	1.11	-------
32	0 Mo			0.16	-------	-------	-------	-------	-------	0.20	-------	-------	-------
33	1 Mo			.16	1.76	0.75	20.46	20.52	21.59	1.00	2.02	1.06	0.15
34	2 Mo			.16	-------	-------	-------	-------	-------	2.34	-------	-------	-------
35	4 Mo			.13	-------	-------	-------	-------	-------	4.99	-------	-------	-------
36	6 Mo			.12	-------	-------	-------	-------	-------	6.99	-------	-------	-------
37	0 W			0.14	-------	-------	-------	-------	-------	-------	0.04	-------	-------
38	1 W			.15	1.74	0.75	20.75	20.50	21.68	3.42	.82	1.02	0.15
39	4 W			.14	-------	-------	-------	-------	-------	-------	4.66	-------	-------
40	6 W			.16	-------	-------	-------	-------	-------	-------	7.13	-------	-------
47	0 Cb			0.14	-------	-------	-------	-------	-------	-------	-------	0.03	-------
48	2 Cb			.14	1.74	0.70	20.65	20.59	20.08	3.20	1.94	1.97	0.14
49	4 Cb			.14	-------	-------	-------	-------	-------	-------	-------	4.07	-------
50	6 Cb			.16	-------	-------	-------	-------	-------	-------	-------	6.09	-------
41	Low N			0.14	-------	-------	-------	-------	-------	-------	-------	-------	0.004
42	0.07 N			.15	-------	-------	-------	-------	-------	-------	-------	-------	.08
81	0.22 N			.15	-------	-------	-------	-------	-------	-------	-------	-------	.18
	Mo	W	Cb										
43	0	0	0	0.12	-------	-------	-------	-------	-------	0.09	0.00	0.00	-------
44	0	0	0	.18	1.72	0.74	20.22	20.62	20.09	.00	.00	.00	0.14
45	2	0	0	.14	-------	-------	-------	-------	-------	2.09	.00	.00	-------
46	4	0	0	.13	-------	-------	-------	-------	-------	4.01	.00	.00	-------
53	0	2	0	.13	-------	-------	-------	-------	-------	.01	2.05	.00	-------
54	0	4	0	.14	-------	-------	-------	-------	-------	.02	3.97	.00	-------
55	2	2	0	0.15	1.71	0.72	20.28	20.41	20.20	2.33	2.01	0.00	0.15
56	4	2	0	.13	-------	-------	-------	-------	-------	4.05	2.04	.00	-------
57	2	4	0	.14	-------	-------	-------	-------	-------	1.98	4.10	.00	-------
58	4	4	0	.13	-------	-------	-------	-------	-------	4.04	4.11	.00	-------
59	2	2	2	0.16	-------	-------	-------	-------	-------	2.19	2.04	1.98	-------
60	4	2	2	.13	-------	-------	-------	-------	-------	4.12	2.07	1.98	-------
61	2	4	2	.15	1.69	0.80	19.99	20.50	20.54	2.32	4.12	2.03	0.14
62	2	2	4	.15	-------	-------	-------	-------	-------	2.02	1.93	4.09	-------
63	2	0	2	0.11	-------	-------	-------	-------	-------	2.10	0.00	1.93	-------
64	2	0	4	.15	-------	-------	-------	-------	-------	2.32	.03	4.23	-------
65	4	0	2	.13	-------	-------	-------	-------	-------	4.02	.00	2.10	-------
66	4	0	4	.13	-------	-------	-------	-------	-------	3.90	.00	4.08	-------
67	4	4	4	.13	1.70	0.71	20.18	20.36	20.12	4.12	3.94	3.94	0.17
77	0	0	1	0.15	-------	-------	-------	-------	-------	-------	-------	0.96	-------
68	0	0	2	.18	-------	-------	-------	-------	-------	0.00	0.00	1.81	-------
69	0	0	4	.14	-------	-------	-------	-------	-------	.00	.00	3.76	-------
70	0	2	2	.16	-------	-------	-------	-------	-------	.01	2.00	1.92	-------
71	0	4	2	.17	-------	-------	-------	-------	-------	.02	3.93	1.91	-------
72	0	2	4	.14	1.80	0.66	20.21	20.39	20.51	.01	2.01	4.18	0.12
73	0	4	4	.15	-------	-------	-------	-------	-------	.02	3.92	4.05	-------
82	2	4	4	0.16	-------	-------	-------	-------	-------	-------	-------	-------	-------
83	4	2	4	.13	-------	-------	-------	-------	-------	3.85	1.85	4.38	-------
84	4	4	2	.14	1.64	0.74	20.18	20.51	19.95	-------	-------	2.25	0.17

[a] Values given only where actual chemical analyses were made; blank spaces indicate same aim value as that of basic alloy.
[b] Alloys 1 to 6 were prepared to develop and standardize melting practice.
[c] Alloys 7, 8, 10, 11, and 12 were prepared to determine control of properties with processing from heat to heat of basic alloy.
[d] Alloy 27, aimed at low Si, resulted in basic value.
[e] Deoxidation practice was varied on alloys 74 and 75 of basic analysis.

TABLE II.—TYPICAL MELTING RECORD

[Alloy 72. Aim analysis, percent: 0.15 C, 1.7 Mn, 0.5 Si, 20 Cr, 20 Ni, 20 Co, 0 Mo, 2 W, 4 Cb, 0.12 N]

(a) Charge

Melting stock	Weight (grams)	Composition contributed by melting stock (percent)										
		Fe	C	Mn	Si	Cr	Ni	Co	Mo	W	Cb	N
Electrolytic nickel	820	------	------	------	------	------	20.5	------	--	------	------	------
Cobalt rondles	800	------	------	------	------	------	------	20.0	--	------	------	------
Armco iron	721	17.99	0.004	0.005	------	------	------	------	--	------	------	------
Ferrochromium (low C, high N)	544	4.27	.011	------	0.053	9.15	------	------	--	------	------	0.110
Ferrochromium (high C, low N)	72	.45	.088	------	.035	1.21	------	------	--	------	------	.001
Ferrochromium (low C, low N)	599	4.41	.008	------	.066	10.64	------	------	--	------	------	.006
Electrolytic manganese	68	------	------	1.69	------	------	------	------	--	------	------	------
Ferrosilicon (50 percent Si)	0	------	------	------	------	------	------	------	--	------	------	------
Ferromolybdenum (60 percent Mo)	0	------	------	------	------	------	------	------	--	------	------	------
Ferrotungsten (80 percent W)	100	0.43	0.012	0.005	0.014	------	------	------	--	2.00	------	------
Ferrocolumbium (60 percent Cb)	276	2.38	.023	------	.464	------	------	------	--	------	4.00	------
Totals	4000	29.93	0.146	1.70	0.63	21.0	20.5	20.0	0	2.00	4.00	0.117
Actual analysis	------	------	0.14	1.80	0.66	20.21	20.39	20.51	0	2.01	4.18	0.12

(b) Melt data

Time (min)	Operation
0	Fe, Cr, Ni, and Co melting stock placed in hot crucible (fifth heat in crucible).
0	Power on, 400 volts, 10 kw.
15	Charge melted.
17	Slag skimmed.
18	Mn added.
20	Ferrotungsten and ferrocolumbium added.
22	Power down to 300 volts, 5 kw.
23	15 grams of 30-percent-Ca and 65-percent-Si alloy added to deoxidize.
24	Slag skimmed.
25	Power off. Bath temperature: Leeds and Northrup optical pyrometer—2360° F. Pt, Pt-Rh immersion thermocouple—2340° F.
27	Heat poured.

Cooling-curve data taken on hot-top metal. Temperature halt in curve at 2495° F.

TABLE III.—MELTING DATA FOR EXPERIMENTAL ALLOYS

Alloy	Modification from basic alloy (percent)			Bath temperature before pouring (°F)		Cooling-curve temperature halt (°F)
				Leeds and Northrup optical pyrometer	Pt, Pt-Rh immersion thermocouple	
7	Basic			----------	2570	----------
8	Basic			----------	2560	----------
10	Basic			----------	2665	----------
11	Basic			----------	2680	----------
12	Basic			----------	2785	----------
27	Basic			2625	2600	2466
74	Basic, no deoxidation			2780	2840	----------
75	Basic, Zr-Si-Fe deoxidant			2730	2745	----------
76	Basic, melted under lime-fluorspar slag.			2525	2520	----------
13	0.08 C			----------	----------	2560
14	0.40 C			----------	2535	----------
15	0.40 C			----------	2535	2500
16	0.60 C			----------	2585	2518
17	0.60 C			----------	2585	>2470
18 [a]	0 Mn			2550	----------	----------
24	0 Mn			2630	2617	>2490
19	0.30 Mn			----------	2640	>2530
20	0.50 Mn			2640	2587	2561
21	1.0 Mn			----------	2560	2555
22	2.5 Mn			----------	2585	2514
79 [a]	Low Si			2675	2682	----------
28	1.2 Si			2640	2723	>2414
80	1.6 Si			2580	2610	2450
51	10 Cr			----------	2758	2542
52	30 Cr			2722	2754	>2430
23	0 Ni			2620	----------	----------
25	10 Ni			2600	2622	2487
26	30 Ni			2690	2634	2456
29	0 Co			2660	2655	2471
30	10 Co			2690	----------	2479
31	32 Co			2670	2657	2459
32	0 Mo			2700	2792	2529
33	1 Mo			2670	2660	2495
34	2 Mo			2660	2643	>2440
35	5 Mo			2710	2684	2471
36	7 Mo			2710	2688	2474
37	0 W			2710	2698	2505
38	1 W			2705	2692	2487
39	5 W			2685	2690	>2490
40	7 W			2700	2693	2484
47	0 Cb			2770	2777	2553
48	2 Cb			----------	2780	2508
49	4 Cb			2650	2703	2464
50	6 Cb			2690	----------	>2390
41	0.004 N			2709	2707	2508
42	.08 N			2720	----------	>2480
81	.18 N			2632	2648	2479
	Mo	W	Cb			
43	0	0	0	2740	2760	2592
44	0	0	0	2780	2790	>2520
45	2	0	0	2750	2768	>2530
46	4	0	0	2750	2770	2537
53	0	2	0	2870	2805	>2520
54	0	4	0	2785	2783	2542
55	2	2	0	2752	2736	2534
56	4	2	0	2742	2762	2529
57	2	4	0	2732	2670	2518
58	4	4	0	2832	2835	2482
59	2	2	2	----------	2790	2484
60	4	2	2	2685	2680	2466
61	2	4	2	2725	2726	2474
62	2	2	4	2710	2703	>2400
63	2	0	2	2802	2847	>2500
64	2	0	4	2740	2792	2460
65	4	0	2	2750	2766	2458
66	4	0	4	2670	2720	2442
67	4	4	4	2650	2678	2424
77	0	0	1	----------	----------	2613
68	0	0	2	2810	2815	2521
69	0	0	4	2710	2740	2476
70	0	2	2	2750	2782	2532
71	0	4	2	2740	2757	>2510
72	0	2	4	2800	2840	2495
73	0	4	4	2730	2773	2476
82	2	4	4	2590	2602	>2400
83	4	2	4	2625	2680	2419
84	4	4	2	2602	2585	2442

[a] Blow hole in ingot of low-Mn (18) and low-Si (79) heats.

TABLE IV.—TYPICAL FORGING RECORD

[Alloy 65, 4Mo-0W-2Cb]

(a) Forging operations

Time	Operation
8:25 a. m.	Ingot moved to center of furnace at 2200° F after a preheat near door of furnace.
8:30	Ingot on temperature, 2200° F.
9:05	60 hammer blows between flat dies on square faces of ingot; forging only top half of ingot.
9:20	55 blows between flat dies on square faces; 10 blows between flat dies on corners.
9:32	60 blows between flat dies on square faces; 10 blows between flat dies on corners.
9:45	55 blows between flat dies on square faces; 10 blows between flat dies on corners to approximately 1.10-in.-square bar.
9:54	70 blows in first swage.
10:04	21 blows in first swage to approximately 0.95 in. round; 46 blows in second swage.
10:12	64 blows in second swage.
10:18	65 blows in second swage.
10:27	40 blows in second swage to approximately 0.75 in. round; forged piece cut off from unforged half of ingot; forged piece recharged to furnace.
10:40	65 blows in third swage.
10:46	47 blows in third swage.
10:54	55 blows in third swage.
11:02	55 blows in third swage.
11:10	48 blows in third swage.
11:16	55 blows in third swage to approximately 0.53 in. round; this piece cut in two, both pieces recharged to furnace for forging in the last swage.
11:24	54 blows on bar C, 59 blows on bar D in last swage.
11:30	53 blows on bar C, 55 blows on bar D in last swage.
11:36	45 blows on bar C, 52 blows on bar D in last swage.
11:41	45 blows on bar C, 48 blows on bar D in last swage.
11:47	55 blows on bar C, 56 blows on bar D in last swage.
11:52	60 blows on bar C, 62 blows on bar D in last swage.
11:57	45 blows on bar C, 46 blows on bar D in last swage.
12:02	40 blows on bar C, 35 blows on bar D in last swage, finishing to 0.40 in. round.
	Finishing temperatures for all forging operations were 1800° F or above as judged by color.

(b) Forging summary

Die	Heatings	Blows
Flat	4	260
First swage	2	91
Second swage	4	215
Third swage	6	325
Last swage	8	Bar C—397 Bar D—413

Total heatings: 16.
Total blows:
Bar C—797.
Bar D—813.
Forging range: 2200° to 1800° F.

Initial size of ingot:
1.42 in. square tapered to 1.04 in. square.
8.60 in. long.
Final size of forged bars:
Bar C: 0.40 in. round by 19.3 in.
Bar D: 0.40 in. round by 21.0 in.
Reduction during forging: 93 percent.

TABLE V.—FORGING DATA FOR EXPERIMENTAL ALLOYS

Alloy	Bar [a]	Modification from basic alloy (percent)			Total heatings	Approximate total blows	Bar size		Estimated forgeability compared with that of basic alloy
							Length (in.)	Diameter (in.) [b]	
7	A	Basic			8	361	25.5	0.42 sq	
7	B				7	267	14	0.50	
8	A	Basic			8	356	25	0.40 sq	
8	D				18	875	26	0.41	
10	A	Basic			9	428	25.5	0.42 sq	
10	D				20	939	29	0.41	
11	B	Basic			3	384	30	0.50 sq	
12	A	Basic			8	434	28	0.41 sq	
27	D	Basic			14	824	25.2	0.43	
74	D	Basic, no deoxidation			18	799	22	0.40	Same.
75	D	Basic, Zr–Si–Fe deoxidant			14	723	21	0.42	Same.
76	D	Basic, melted under slag			------	------	------	------	Poorer—many cracks during initial flat die work; forging discontinued.
13	A	0.08 C			5	292	24.5	0.40 sq	Better—more plastic.
15	A	0.40 C			8	350	21.5	0.42 sq	Poorer—corner and end cracks; less plastic.
16	A	0.60 C			5	169	9.5	0.51	Poorer—many center and corner cracks; less plastic.
16	B				5	271	13	0.42 sq	
24	D	0 Mn			15	909	20.5	0.41	Poorer—many bad corner cracks developed during forging on flat dies.
19	A	0.30 Mn			5	345	23.5	0.44 sq	Same—some corner cracks.
20	A	0.50 Mn			6	460	27.5	0.40 sq	Same—some end cracks.
21	A	1.0 Mn			6	444	29	0.41 sq	Same.
22	A	2.5 Mn			4	344	25	0.41 sq	Slightly better.
28	D	1.2 Si			15	895	26.4	0.44	Same.
80	D	1.6 Si			15	863	23	0.41	Same.
51	D	10 Cr			15	706	21	0.40	Better—much more plastic.
52	D	30 Cr			20	959	21.5	0.40	Poorer—much less plastic.
23	D	0 Ni			12	613	15.4	0.47	Better—more plastic.
25	D	10 Ni			11	597	17	0.48	Better—more plastic.
26	D	30 Ni			16	846	22	0.45	Poorer—less plastic.
29	D	0 Co			12	740	25.2	0.42	Better—more plastic.
30	D	10 Co			15	813	27.2	0.41	Same.
31	D	32 Co			13	724	20.5	0.41	Poorer—less plastic.
32	D	0 Mo			14	771	22	0.41	Same.
33	D	1 Mo			18	660	22	0.42	Same.
34	D	2 Mo			19	928	27.2	0.42	Same.
35	D	5 Mo			16	804	22.5	0.40	Same.
36	D	7 Mo			16	818	23	0.41	Same.
37	D	0 W			11	603	17	0.41	Better—more plastic.
38	D	1 W			14	674	21.5	0.41	Better—more plastic.
39	D	5 W			14	750	21.5	0.41	Poorer—less plastic.
40	D	7 W			17	874	22	0.42	Poorer—less plastic than 39.
47	D	0 Cb			16	629	17.5	0.41	Poorer—many cracks developed during forging on flat dies.
48	D	2 Cb			16	736	20.3	0.40	Same.
49	D	4 Cb			19	815	21	0.41	Poorer—less plastic.
50	D	6 Cb			18	889	21	0.40	Poorer—less plastic; no forging cracks.
41	D	0.004 N			14	684	20.5	0.41	Same.
42	D	0.08 N			16	770	19.5	0.41	Same.
81	D	0.18 N			15	812	21.8	0.41	Same.
		Mo	W	Cb					
43	D	0	0	0	16	854	26	0.45	Better—much more plastic.
45	D	2	0	0	14	646	22.5	0.41	Poorer—more plastic but more subject to cracking.
46	D	4	0	0	17	700	22	0.41	Poorer—similar to 45.
53	D	0	2	0	15	683	22.5	0.40	Poorer—more plastic but much more subject to cracking.
54	D	0	4	0	18	821	23	0.41	Poorer—same as 53.
55	D	2	2	0	19	1050	28.8	0.40	Poorer—much more tendency to crack.
56	D	4	2	0	19	1034	28.5	0.41	Poorer—same as 55.
57	D	2	4	0	20	881	21	0.41	Poorer—less plastic; tendency to crack.
58	D	4	4	0	22	998	21.5	0.40	Poorer—same as 57.
59	D	2	2	2	19	904	24	0.41	Same.
60	D	4	2	2	18	894	22.5	0.41	Same.
61	D	2	4	2	18	826	21.5	0.40	Poorer—less plastic.
62	D	2	2	4	19	917	23	0.40	Poorer—same as 61.
63	D	2	0	2	14	728	20.5	0.40	Better—more plastic.
64	D	2	0	4	14	764	19	0.40	Same.
65	D	4	0	2	16	813	21	0.40	Same.
66	D	4	0	4	16	859	21.5	0.40	Poorer—less plastic.
67	D	4	4	4	12	674	18.3	0.40	Poorer—less plastic.
77	D	0	0	1	15	841	26.5	0.40	Better—more plastic.
68	D	0	0	2	13	687	21	0.40	Better—more plastic.
69	D	0	0	4	10	521	17.3	0.41	Better—more plastic.
70	D	0	2	2	17	782	21	0.40	Same.
71	D	0	4	2	17	850	24	0.40	Same.
72	D	0	2	4	16	771	21.5	0.40	Same.
73	D	0	4	4	16	822	22.5	0.40	Same.
82	D	2	4	4	16	833	24	0.41	Poorer—less plastic.
83	D	4	2	4	16	850	22	0.41	Poorer—same as 82.
84	D	4	4	2	14	851	20.8	0.41	Poorer—same as 82.

[a] Letter designates location of bar in ingot as follows:
Bar A was from bottom quarter of ingot in all cases except 16A, which was from lower middle quarter of ingot
Bar B was from lower middle quarter except 16B, which was from bottom of ingot
Bar D was from upper quarter of ingot

[b] All bars were rounds except where squares (sq) are indicated.

TABLE VI.—INFLUENCE OF FORGING AND MELTING VARIABLES ON RUPTURE TEST CHARACTERISTICS AT 1200° F OF BASIC ALLOY [a]

[Heat treatment: 2200° F, 1 hr, water-quenched; 1400° F, 24 hr, air-cooled]

Alloy	Rupture strength (psi)		Estimated 100-hr rupture elongation (percent in 1 in.)	Stress (psi) to cause creep rates of—		Minimum creep rate at 40,000 psi (percent/hr)
	100 hr	1000 hr		0.01 percent/hr	0.1 percent/hr	
Final adopted forging procedure—round bar stock						
8	48,500	37,000	22	35,800	47,500	0.022
10	50,000	38,000	19	35,400	49,000	.023
27	48,000	37,000	25	37,000	46,200	.023
Average	48,800	37,300	22	36,000	47,600	0.023
Range	48,000 to 50,000	37,000 to 38,000	19 to 25	35,400 to 37,000	46,200 to 49,000	0.022 to .023
Preliminary forging procedure—square bar stock						
7	48,500	37,000	20	35,800	46,300	0.028
8	46,500	[b] 36,000	18	34,500	46,000	.033
10	52,000	36,500	20	35,400	50,100	.022
11	48,000	38,000	22	36,000	47,500	.024
12	49,000	38,500	22	38,200	46,000	.018
Average	48,800	37,200	20	36,000	47,200	0.025
Range	46,500 to 52,000	36,000 to 38,500	18 to 22	34,500 to 38,200	46,000 to 50,100	0.018 to .033
Variable deoxidation practice [c]—round bar stock						
74 (none)	52,000	[b] 41,000	16	[b] 40,700	[b] 55,500	[b] 0.0090
75 (Zr-Si-Fe)	53,000	40,500	18	42,300	57,000	[b] .0065
Comparative properties of commercial bar stock of basic alloy						
Heat 30276	50,000	[b] 42,000	14	----------	----------	----------
A–1726	47,000	[b] 42,000	16	----------	----------	----------

[a] Results based on detailed rupture test data given in table X.
[b] Estimated value.
[c] Deoxidation practice:
None—no deoxidation
Zr-Si-Fe—zirconium-silicon-iron deoxidant

TABLE VII.—INFLUENCE OF CHEMICAL COMPOSITION ON MICROSTRUCTURAL CLASSIFICATIONS OF MODIFIED ALLOYS

Individual variation of elements in basic alloy						Simultaneous variation of Mo, W, and Cb																							
						Variable Mo								Variable W								Variable Cb							
Alloy	Alloy modification (percent)	Microstructutal classification [a]				Alloy	Alloy modification (percent)			Microstructural classification [a]				Alloy	Alloy modification (percent)			Microstructural classification [a]				Alloy	Alloy modification (percent)			Microstructural classification [a]			
		IC	GS	GB	MP		Mo	W	Cb	IC	GS	GB	MP		Mo	W	Cb	IC	GS	GB	MP		Mo	W	Cb	IC	GS	GB	MP
Basic		II	5	y	Rb	43	0	0	0	I	1	z	Cb	43	0	0	0	I	1	z	Cb	43	0	0	0	I	1	z	Cb
13	0.08 C	II	5	y	Ra	45	2	0	0	I	3	z	Cb	53	0	2	0	I	1	z	Cc	77	0	0	1	II	6	y	Ra
Basic	0.15 C	II	5	y	Rb	46	4	0	0	I	2	y	Cc	54	0	4	0	I	2	z	Cc	68	0	0	2	II	5	y	Rb
15	0.40 C	III	6	y	Rc																	69	0	0	4	IV	6	y	Rb
16	0.60 C	IV	8	y	Rd																								
24	0 Mn	II	5	y	Rb	53	0	2	0	I	1	z	Cc	45	2	0	0	I	3	z	Cb	45	2	0	0	I	3	z	Cb
19	0.80 Mn	II	5	y	Rb	55	2	2	0	I	1	z	Cc	55	2	2	0	I	1	z	Cc	63	2	0	2	II	5	y	Rb
20	0.50 Mn	II	5	y	Rb	47	3	2	0	I	1	z	Cc	57	2	4	0	I	2	z	Cc	64	2	0	4	IV	5	y	Rb
21	1.0 Mn	II	5	y	Rb	56	4	2	0	I	1	y	Cc																
Basic	1.7 Mn	II	5	y	Rb																								
22	2.5 Mn	II	5	y	Rb																								
Basic	0.5 Si	II	5	y	Rb	68	0	0	2	II	5	y	Rb	68	0	0	2	II	5	y	Rb	53	0	2	0	I	1	z	Cc
28	1.2 Si	II	5	y	Rb	63	2	0	2	II	5	y	Rb	70	0	2	2	II	5	y	Rb	70	0	2	2	II	5	y	Rb
30	1.6 Si	II	6	y	Rb	65	4	0	2	II	5	y	Rb	71	0	4	2	II	4	y	Rb	72	0	2	4	IV	7	y	Rb
51	10 Cr	II	6	x	Ra																								
Basic	20 Cr	II	5	y	Rb																								
52	60 Cr	II	3	y	Cd	70	0	2	2	II	5	y	Rb	63	2	0	2	II	5	y	Rb	55	2	2	0	I	1	z	Cc
23	0 Ni	IV [b]	6	x	Rb [b]	59	2	2	2	III	6	y	Rb	59	2	2	2	III	6	y	Rb	54	2	2	1	II	5	y	Rb
25	10 Ni	II	4	y	Rb	48	3	2	2	III	6	y	Rb	61	2	4	2	III	5	y	Rb	59	2	2	2	III	6	y	Rb
Basic	20 Ni	II	5	y	Rb	60	4	2	2	III	7	y	Rb									62	2	2	4	IV	6	y	Rb
26	30 Ni	II	5	y	Rb																								
29	0 Co	II	6	y	Rd	69	0	0	4	IV	6	y	Rb	69	0	0	4	IV	6	y	Rb	54	0	4	0	I	2	z	Cc
30	10 Co	II	5	y	Rb	64	2	0	4	IV	5	y	Rb	72	0	2	4	IV	7	y	Rb	71	0	4	2	II	4	y	Rb
Basic	20 Co	II	5	y	Rb	66	4	0	4	IV	7	y	Rc	73	0	4	4	IV	6	y	Rc	73	0	4	4	IV	6	y	Rc
31	32 Co	II	1–5	y	Rb																								
32	0 Mo	II	4	y	Rb																								
33	1 Mo	II	2–6	y	Rb	72	0	2	4	IV	7	y	Rb	64	2	0	4	IV	5	y	Rb	57	2	4	0	I	2	z	Cc
34	2 Mo	II	5	y	Rb	62	2	2	4	IV	6	y	Rb	62	2	2	4	IV	6	y	Rb	61	2	4	2	III	5	y	Rb
Basic	3 Mo	II	5	y	Rb	49	3	2	4	IV	8	x	Rd	82	2	4	4	IV	8	y	Rd	82	2	4	4	IV	8	y	Rd
35	5 Mo	II	4–5	y	Rb	83	4	2	4	IV	8	y	Rd																
36	7 Mo	II	4–5	y	Rb																								
37	0 W	II	5	y	Rb	54	0	4	0	I	2	z	Cc	46	14	0	0	I	2	y	Cc	46	4	0	0	I	2	y	Cc
38	1 W	II	5	y	Rb	57	2	4	0	I	2	z	Cc	56	4	2	0	I	1	y	Cc	65	4	0	2	II	5	y	Rb
Basic	2 W	II	5	y	Rb	58	4	4	0	I	3	z	Cc	58	4	4	0	I	3	z	Cc	66	4	0	4	IV	7	y	Rc
39	5 W	II	5	y	Rb																								
40	7 W	II	5	y	Rb																								
47	0 Cb	I	1	z	Cc																								
Basic	1 Cb	II	5	y	Rb	71	0	4	2	II	4	y	Rb	65	4	0	2	II	5	y	Rb	56	4	2	0	I	1	y	Cc
48	2 Cb	III	6	y	Rb	61	2	4	2	III	5	y	Rb	60	4	2	2	III	7	y	Rb	60	4	2	2	III	7	y	Rb
49	4 Cb	IV	8	x	Rd	84	4	4	2	III	6	y	Rb	84	4	4	2	III	6	y	Rb	83	4	2	4	IV	8	y	Rd
50	6 Cb	IV	8	x	Rd																								
41	0.004 N	II	5	y	Rb	78	0	4	4	IV	6	y	Rc	66	4	0	4	IV	7	y	Rc	58	4	4	0	I	3	z	Cc
42	0.08 N	II	5	y	Rb	82	2	4	4	IV	8	y	Rd	83	4	2	4	IV	8	y	Rd	84	4	4	2	III	6	y	Rb
Basic	0.15 N	II	5	y	Rb	67	4	4	4	IV	8	y	Rd	67	4	4	4	IV	8	y	Rd	67	4	4	4	IV	8	y	Rd
31	0.18 N	II	5	y	Rb																								

[a] Classification key:

Solution-treated structure	Aged structure			
Amount of insoluble constituent (IC)	A. S. T. M. grain size number (GS)	Amount of grain boundary precipitate (GB)	Aging precipitate in matrix (MP)	
			Type	Amount
I—small. II—medium. III—large. IV—very large.	1 (up to $1\frac{1}{2}$ grains/sq in.) to 8 (96 grains or more/sq in.)	x—small. y—medium. z—large.	C—precipitate tends to follow crystallographic planes. R—random precipitate.	a—small. b—medium. c—large. d—very large.

[b] Ferrite-sigma-type phase in 0-percent-Ni alloy 23. A large amount of precipitate occurred in sigma phase during aging but only a small amount in austenite matrix.

TABLE VIII.—SUMMARY OF INFLUENCE OF ALLOYING ELEMENTS ON INCREASING TENDENCY FOR MICROSTRUCTURAL CHANGES

Alloying element varied	Composition range (percent)	Insoluble constituent increase	Grain size decrease	Grain boundary precipitation increase	Mode of matrix precipitation	Amount of matrix precipitation increase
C	0.08–0.60	Strong	Mild	None	Random	Very strong.
Mn	0–2.5	None	None	None	Random	None.
Si	0.5–1.6	None	Mild	None	Random	None.
Cr	10–20	None	Negative mild	Mild	Random	Mild.
Cr	20–30	None	Negative mild	None	Random to preferred	Strong.
Ni	0–10	Negative strong [a]	Negative mild	Negative weak	Random	None.[a]
Ni	10–30	None	Weak	None	Random	None.
Co	0–32	None	Negative mild	None	Random	Negative mild.
Mo	0–4	None	Weak	None	Random [b]	Weak.
W	0–4	None	Weak	None	Random [b]	Weak.
Cb	0–1	Strong	Strong	Negative mild	Preferred to random	Negative mild.
Cb	1–4	Strong	Mild	None	Random	Mild.
N	0.004–0.18	None	None	None	Random	None.

[a] Ferrite-sigma phase in 0-percent-Ni alloy in all conditions. A large amount of aging precipitate occurred in sigma phase, but only a small amount in austenite matrix.
[b] Mode of precipitation not affected by Mo and W. In alloys containing Cb precipitate was random; without Cb precipitate followed a preferred orientation.

TABLE IX.—HARDNESS DATA ON EXPERIMENTAL ALLOYS

Alloy	Modification from basic alloy (percent)[a]			Vickers hardness number[b]	
				Solution-treated; 2200° F, 1 hr, water-quenched	Solution-treated and aged; 1400° F, 24 hr, air-cooled
7	Basic (sq)			229	245
8	Basic (sq)			229	243
8	Basic			229	247
10	Basic (sq)			237	272
10	Basic			234	259
11	Basic (sq)			235	247
12	Basic (sq)			229	252
27	Basic			211	221
74	Basic, no deoxidation			214	223
75	Basic, Zr-Si-Fe deoxidant			229	247
13	0.08 C (sq)			206	219
15	0.40 C (sq)			261	288
16	0.60 C (sq)			290	309
24	0 Mn			227	251
19	0.30 Mn (sq)			227	251
20	0.50 Mn (sq)			223	241
21	1.0 Mn (sq)			216	231
22	2.5 Mn (sq)			229	245
28	1.2 Si			221	249
80	1.6 Si			243	242
51	10 Cr			177	193
52	30 Cr			283	408
23	0 Ni			288	330
25	10 Ni			238	261
26	30 Ni			226	252
29	0 Co			210	252
30	10 Co			205	226
31	32 Co			221	235
32	0 Mo			203	219
33	1 Mo			202	210
34	2 Mo			197	211
35	5 Mo			205	233
36	7 Mo			221	245
37	0 W			205	218
38	1 W			206	223
39	5 W			223	245
40	7 W			243	254
47	0 Cb			176	219
48	2 Cb			206	238
49	4 Cb			258	325
50	6 Cb			229	296
41	0.004 N			197	208
42	0.08 N			233	254
81	0.18 N			234	261
	Mo	W	Cb		
43	0	0	0	157	190
45	2	0	0	182	205
46	4	0	0	185	235
53	0	2	0	176	210
54	0	4	0	187	235
55	2	2	0	201	242
56	4	2	0	194	243
57	2	4	0	198	251
58	4	4	0	202	252
59	2	2	2	213	220
60	4	2	2	234	252
61	2	4	2	234	242
62	2	2	4	226	240
63	2	0	2	192	215
64	2	0	4	215	238
65	4	0	2	221	229
66	4	0	4	235	258
67	4	4	4	258	311
77	0	0	1	197	204
68	0	0	2	196	201
69	0	0	4	204	216
70	0	2	2	211	213
71	0	4	2	223	227
72	0	2	4	215	219
78	0	4	4	221	229
82	2	4	4	237	258
83	4	2	4	242	288
84	4	4	2	245	272

[a] All tests were made on round bar stock except where square bar stock is indicated (sq).
[b] Vickers hardness tests were made with a 50-kg load on bar-stock cross sections of metallographic specimens.

TABLE X—RUPTURE TEST CHARACTERISTICS AT 1200° F FOR MODIFIED ALLOYS

[Heat treatment: 2200° F, 1 hr, water-quenched; 1400° F, 24 hr, air-cooled]

Alloy	Alloy modification (percent) [a]	Stress (psi)	Time (hr)	Elongation in 1 in. (percent)	Reduction of area (percent)	Minimum creep rate (percent/hr)	Rupture strength (psi)		Estimated 100-hr rupture elongation (percent in 1 in.)	Stress (psi) to cause creep rates of—		Minimum creep rate at 40,000 psi (percent/hr)
							100 hr	1000 hr		0.01 percent/hr	0.1 percent/hr	
7	Basic (square)	50,000	74	20	26.8	———	48,500	37,000	20	35,800	46,300	0.028
		45,000	184	34	39.2	0.076						
		40,000	516	27	37.4	.028						
	(Round)	50,000	82	38	34.8	———	49,000	[b] 37,000	30	———	———	———
		45,000	209	31	31.5	.070						
8	Basic (square)	50,000	51	19	17.8	———	46,500	[b] 36,000	18	34,500	46,000	.033
		45,000	142	17	20.5	.083						
		40,000	368	21	15.4	.033						
	(Round)	50,000	87	22	18.3	.160	48,500	37,000	22	35,800	47,500	.022
		45,000	162	23	22.6	.066						
		40,000	499	25	23.4	.022						
		37,000	1175	37	38.4	.0124						
10	Basic (square)	52,000	98	20	26.1	.115	52,000	36,500	20	35,400	50,100	.022
		50,000	155	21	21.1	.083						
		45,000	259	27	30.1	.054						
		40,000	422	21	29.5	.022						
	(Round)	53,000	61	15	13.8	———	50,000	38,000	19	35,400	49,000	.023
		50,000	100	19	24.0	.110						
		45,000	240	36	37.0	.057						
		40,550	584	24	27.5	.023						
11	Basic (square)	50,000	77	26	24.0	.112	48,000	38,000	22	36,000	47,500	.024
		45,000	170	19	22.6	.064						
		40,000	575	28	26.2	.024						
12	Basic (square)	50,000	88	22	28.4	———	49,000	38,500		38,200	46,000	.018
		45,000	209	25	36.6	.070						
		40,000	719	22	30.9	.018						
27	Basic (round)	50,000	82	26	25.5	———	48,000	37,000	25	37,000	46,200	.023
		45,000	176	18	16.8	.075						
		40,000	525	23	26.5	.023						
74	Basic, no deoxidation	57,000	33	22	21.3	———	52,000	[b] 41,000	16	[b] 40,700	[b] 55,500	[b] 0.0090
		54,000	98	16	15.5	———						
		49,000	184	15	18.4	0.039						
		45,000	417	14	14.4	.021						
75	Basic, Zr–Si–Fe deoxidant	57,000	64	16	15.5	———	55,000	40,500	18	42,300	57,000	[b] .0065
		54,000	134	18	21.3	.075						
		49,000	268	18	19.1	.025						
		45,000	502	20	19.9	.019						
13	0.08 C (square)	50,000	79	14	21.3	———	48,500	[b] 36,000	14	[b] 36,000	[b] 47,000	[b] 0.033
		45,000	222	14	10.9	0.040						
		40,000	325	26	21.8	.033						
15	0.40 C (square)	50,000	106	19	21.1	———	50,500	36,500	19	39,500	49,000	.0102
		45,000	193	19	16.2	.040						
		40,000	518	9	12.4	.0102						
16	0.60 C (square)	50,000	39	18	18.4	———	46,000	37,500	15	38,000	[b] 49,000	[b] .016
		45,000	111	13	12.4	.090						
		40,000	435	12	16.2	.018						
	(Round)	49,734	78	17	16.1	———	48,000	37,000	15	———	———	———
		45,000	178	14	16.2	.036						
		40,000	528	13	15.4	.0145						
24	0 Mn (round)	54,000	64	8	28.8	———	50,000	[b] 36,000	13	[b] 39,000	———	[b] 0.013
		50,000	125	13	10.9	———						
		45,000	195	10	8.7	0.036						
19	0.30 Mn (square)	53,000	43	22	28.4	———	49,000	[b] 41,000	24	[b] 39,000	49,000	[b] .013
		50,000	83	24	22.6	.140						
		45,000	352	19	21.8	.032						
20	0.50 Mn (square)	50,000	67	22	21.9	———	48,000	[b] 37,000	22	36,000	51,000	.019
		45,000	246	21	22.6	.042						
		40,000	452	23	20.5	.019						
21	1.0 Mn (square)	50,000	53	21	19.9	———	47,000	38,000	20	39,000	47,000	.015
		45,000	199	14	19.7	.059						
		40,000	617	16	17.6	.0145						
22	2.5 Mn (square)	50,000	85	20	18.4	.147	49,000	37,500	20	35,000	48,000	.024
		45,000	208	19	23.4	.065						
		40,000	563	26	24.0	.024						
28	1.2 Si	50,000	41	26	20.5	———	45,000	34,500	28	33,700	43,000	0.051
		45,000	115	28	30.9	0.145						
		40,000	224	31	26.8	.051						
		36,000	672	26	35.5	.0188						
80	1.6 Si	50,000	28	41	37.3	———	43,500	[b] 34,000	40	———	43,000	.071
		45,000	53	42	36.0	———						
		43,000	114	38	34.2	.102						
		40,000	208	40	42.3	.071						
51	10 Cr	45,180	16	25	24.8	———	39,000	[b] 32,000	25	[b] 30,000	38,000	0.160
		42,500	36	25	27.5	———						
		40,000	109	25	27.5	0.16						
		35,000	339	21	21.8	.046						
52	30 Cr	55,000	52	10	11.0	———	52,000	42,500	10	42,800	[b] 57,000	.006
		50,000	152	8	10.2	.035						
		45,000	264	6	12.5	.0160						
		45,000	518	17	17.6	.0134						
23	0 Ni	50,000	38	13	13.1	———	46,000	34,500	25	34,000	46,000	0.037
		49,000	37	25	24.8	———						
		45,000	133	26	24.0	0.088						
		40,000	305	25	24.8	.037						
		35,000	855	28	25.6	.0126						
25	10 Ni	50,000	86	17	14.7	———	49,000	38,000	17	39,200	51,000	.012
		45,000	217	15	14.6	.034						
		40,000	642	14	15.4	.012						
26	30 Ni	50,000	56	24	22.6	———	47,000	[b] 35,500	25	[b] 32,500	45,000	.044
		45,000	152	27	24.8	.100						
		40,000	369	29	26.2	.044						

[a] Square, forged as square bar; round, forged as round bar: all heats higher than 22 were forged as round bars only.
[b] Estimated.

TABLE X.—RUPTURE TEST CHARACTERISTICS AT 1200° F FOR MODIFIED ALLOYS—Continued

[Heat treatment: 2200° F, 1 hr, water-quenched; 1400° F, 24 hr, air-cooled]

Alloy	Alloy modification (percent) [a]			Stress (psi)	Time (hr)	Elongation in 1 in. (percent)	Reduction of area (percent)	Minimum creep rate (percent/hr)	Rupture strength (psi)		Estimated 100-hr rupture elongation (percent in 1 in.)	Stress (psi) to cause creep rates of—		Minimum creep rate at 40,000 psi (percent/hr)
									100 hr	1000 hr		0.01 percent/hr	0.1 percent/hr	
29	0 Co			45,000	89	19	22.6	0.130	44,500	36,000	20	34,500	43,500	0.051
				40,000	278	18	27.5	.051						
				36,000	1126	30	31.5	.0131						
30	10 Co			45,000	86	14	24.0	.185	43,000	35,000	20	34,000	43,000	.043
				40,000	142	25	24.6	.043						
				35,000	950	26	31.5	.015						
31	32 Co			50,000	69	14	16.8	.143	48,000	39,000	12	39,000	49,000	.013
				45,000	173	9	13.1	.039						
				40,000	280	8	12.5	.0122						
				40,000	(c)	-------	-------	.011						
				40,000	719	23	22.6	.0148						
32	0 Mo			50,000	11	23	24.0	-------	41,500	[b] 34,000	18	35,200	41,500	0.062
				44,000	50	19	21.1	-------						
				40,000	168	17	19.7	0.062						
				37,000	352	14	19.7	.019						
33	1 Mo			50,000	16	13	10.9	-------	42,000	[b] 33,500	10	35,000	44,300	.037
				44,000	32	11	11.7	.088						
				40,000	129	9	12.5	.037						
				37,000	357	12	17.6	.018						
34	2 Mo			50,000	49	9	15.5	-------	44,500	[b] 34,500	15	[b] 36,000	[b] 44,000	.023
				45,000	64	11	15.5	-------						
				44,000	55	11	13.8	-------						
				42,000	156	16	16.7	.055						
				40,000	243	8	19.0	.023						
35	5 Mo			52,500	58	21	22.6	-------	48,500	[b] 38,000	20	[b] 37,000	48,000	.021
				50,000	72	10	15.4	.145						
				45,000	203	20	19.7	.069						
36	7 Mo			55,000	25	16	16.2	-------	50,000	[b] 42,000	25	[b] 38,000	50,000	.010
				51,000	65	26	24.8	-------						
				50,000	157	25	26.8	.079						
				45,000	210	19	21.8	.048						
				45,000	302	21	20.5	.040						
37	0W			50,000	42	19	24.8	-------	45,000	[b] 33,500	21	[b] 34,000	[b] 44,500	0.051
				45,000	102	21	24.0	0.076						
				40,000	248	25	29.5	.051						
38	1W			50,000	35	24	24.0	-------	45,000	[b] 37,500	23	[b] 35,500	[b] 46,500	.027
				45,000	102	23	23.4	.115						
				40,000	443	24	28.8	.027						
39	5W			54,000	50	16	20.5	-------	50,000	[b] 39,000	22	[b] 38,000	[b] 50,500	[b] .015
				50,000	95	22	28.2	.20						
				45,000	270	25	24.0	.023						
40	7W			55,000	62	17	24.0	-------	52,000	[b] 40,000	22	[b] 38,000	50,500	.015
				50,000	156	25	21.8	.078						
				45,000	336	19	14.7	.040						
47	0 Cb			50,000	27.5	9	13.8	-------	40,000	[b] 29,500	12	33,500	44,500	0.045
				45,000	44	11	12.5	-------						
				38,000	161	12	10.9	0.028						
				34,000	376	9	9.5	.011						
48	2 Cb			50,000	51	22	24.0	-------	46,000	[b] 34,000	20	35,500	44,500	.036
				45,000	115	20	25.5	.122						
				40,000	278	26	25.4	.036						
49	4 Cb			55,000	39	32	33.0	-------	49,000	[b] 37,500	30	36,000	48,000	.022
				50,000	112	30	32.3	.133						
				45,000	217	35	34.2	.059						
50	6 Cb			50,000	57	24	22.6	-------	47,000	36,000	25	35,000	46,500	.030
				45,000	178	23	24.0	.070						
				41,000	282	25	23.4	.045						
				39,000	502	27	23.4	.023						
41	0.004 N			50,000	39	24	25.4	-------	45,500	[b] 36,500	25	[b] 35,000	[b] 46,500	0.030
				45,000	110	27	28.2	-------						
				40,000	372	28	30.1	0.030						
42	0.08 N			55,000	55	19	19.7	-------	51,000	[b] 40,000	22	[b] 38,000	50,000	[b] .015
				50,000	190	21	22.6	-------						
				48,000	145	17	21.8	.068						
				45,000	352	24	28.8	.041						
81	0.18 N			54,000	47	19	19.7	-------	49,500	[b] 39,000	20	[b] 38,000	[b] 49,000	[b] .015
				52,000	75	12	19.0	.190						
				50,000	75	16	18.3	.160						
				45,000	280	29	30.9	.038						
	Mo	W	Cb											
43	0	0	0	35,000	11	8	13.9	-------	26,000	19,500	7	25,500	-------	-------
				25,000	141	5	7.2	0.0089						
				20,000	819	13	7.1	.0018						
45	2	0	0	45,000	47	10	10.9	-------	39,000	27,500	9	33,000	[b] 44,000	0.050
				40,000	86	9	9.4	-------						
				35,000	183	7	11.0	.016						
				30,000	578	8	9.4	.005						
46	4	0	0	45,000	65	12	13.8	.126	42,000	[b] 30,500	11	34,100	45,000	.032
				40,000	148	10	13.8	.032						
				35,000	385	8	9.4	.0125						
53	0	2	0	40,000	22	11	10.2	-------	34,000	26,000	7	30,000	37,700	.20
				35,000	88	7	8.7	.050						
				30,000	256	7	5.6	.0098						
				28,000	539	6	5.6	.0050						
54	0	4	0	45,000	31	9	8.0	-------	41,000	[b] 32,000	5	38,200	[b] 50,000	.015
				42,500	72	6	9.4	.022						
				40,000	160	5	6.4	.015						
				35,000	487	2	1.6	.0043						

[a] Square, forged as square bar; round, forged as round bar; all heats higher than 22 were forged as round bars only.
[b] Estimated.
[c] Overheated at 300 hr.

TABLE X.—RUPTURE TEST CHARACTERISTICS AT 1200° F FOR MODIFIED ALLOYS—Concluded

[Heat treatment: 2200° F, 1 hr, water-quenched; 1400° F, 24 hr, air-cooled]

Alloy	Alloy modification (percent) [a]			Stress (psi)	Time (hr)	Elongation in 1 in. (percent)	Reduction of area (percent)	Minimum creep rate (percent/hr)	Rupture strength (psi)		Estimated 100-hr rupture elongation (percent in 1 in.)	Stress (psi) to cause creep rates of—		Minimum creep rate at 40,000 psi (percent/hr)
									100 hr	1000 hr		0.01 per cent/hr	0.1 per-cent/hr	
	Mo	W	Cb											
55	2	2	0	45,000	57	7	6.4	———	41,000	[b] 32,000	6	36,000	[b] 43,500	0.036
				40,000	97	6	8.0	0.036						
				35,000	449	5	8.0	.0070						
56	4	2	0	50,000	43	13	16.8	———	47,000	38,000	15	39,000	47,500	.013
				45,000	154	16	13.8	.056						
				40,000	603	12	16.8	.013						
57	2	4	0	45,000	87	6	10.2	.033	44,000	[b] 33,000	6	40,000	[b] 49,000	.0145
				40,000	213	6	7.1	.0145						
				37,000	418	7	5.6	.0042						
58	4	4	0	54,000	62	10	13.1	———	51,000	40,000	10	40,000	55,000	.010
				50,000	131	9	15.5	.048						
				45,000	232	12	13.1	.024						
				42,000	677	17	20.5	.0137						
59	2	2	2	50,000	63	24	18.3	———	47,000	[b] 36,000	23	[b] 37,000	44,000	0.030
				48,000	85	26	24.0	———						
				45,000	105	23	24.0	0.135						
				40,000	392	19	18.3	.030						
60	4	2	2	50,000	69	27	31.5	———	49,000	[b] 41,000	30	[b] 40,500	47,500	[b] .0090
				48,000	177	30	27.5	.117						
				45,000	241	39	30.8	.047						
61	2	4	2	50,000	69	19	21.3	———	48,500	[b] 40,000	20	[b] 37,500	[b] 49,000	[b] .018
				45,000	277	21	25.5	.048						
62	2	2	4	50,000	51	27	20.5	———	46,000	[b] 34,000	25	[b] 38,500	45,000	.028
				45,000	122	25	25.5	.100						
				40,950	247	31	25.5	.034						
63	2	0	2	45,000	70	34	35.6	———	43,500	[b] 34,000	30	33,600	43,500	0.048
				40,000	200	25	30.1	0.048						
				37,000	462	23	27.5	.024						
64	2	0	4	50,000	26	29	28.8	———	44,000	[b] 34,000	25	———	41,000	.085
				45,000	75	21	24.0	———						
				40,000	236	25	28.2	.085						
65	4	0	2	50,000	43.5	27	26.8	———	45,500	[b] 37,000	30	[b] 35,000	44,000	.030
				45,000	101	30	30.1	.160						
				40,000	444	38	34.8	.030						
66	4	0	4	50,000	46	33	33.0	———	46,000	[b] 36,500	33	[b] 35,000	[b] 45,000	.034
				45,500	114	33	35.6	———						
				40,000	446	43	42.3	.034						
67	4	4	4	54,000	64	37	33.5	———	50,000	38,000	30	36,000	47,500	.025
				50,000	105	28	30.9	.200						
				45,000	253	32	33.8	.052						
				41,000	536	31	32.2	.029						
77	0	0	1	45,000	66	18	16.8	0.160	42,000	[b] 31,000	18	[b] 30,000	42,500	0.065
				40,000	181	15	16.8	.065						
				35,000	357	18	16.8	.030						
68	0	0	2	43,000	48	41	43.7	———	39,500	[b] 31,500	35	[b] 31,500	38,000	.19
				40,000	91	35	34.2	.19						
				35,000	360	27	27.5	.037						
69	0	0	4	42,000	47	31	30.9	———	38,500	[b] 30,500	25	[b] 31,500	37,500	.23
				40,000	76	31	27.5	.23						
				38,000	95	23	24.8	.140						
				35,000	263	24	32.2	.040						
70	0	2	2	44,000	42	24	21.8	———	40,000	[b] 32,000	20	32,800	39,200	0.120
				40,000	102	20	21.9	0.120						
				35,000	424	15	20.5	.023						
71	0	4	2	48,000	47	19	19.0	———	44,000	[b] 37,000	20	38,000	43,800	.026
				45,000	63	17	18.4	.160						
				40,000	368	21	19.7	.026						
72	0	2	4	45,000	60	32	31.5	———	42,000	[b] 32,500	30	[b] 34,000	40,500	.039
				40,000	181	27	28.4	.069						
				37,000	302	28	30.1	.039						
73	0	4	4	48,000	46	22	21.8	———	45,000	[b] 36,500	24	38,000	43,500	.021
				45,000	99	24	24.8	.155						
				40,000	356	17	22.6	.021						
82	2	4	4	50,000	73	20	21.9	0.200	48,000	[b] 37,000	22	35,000	46,000	0.031
				45,000	196	25	25.4	.083						
				40,000	495	32	34.8	.031						
83	4	2	4	50,000	81	32	33.5	———	48,000	[b] 36,000	30	[b] 34,000	44,500	.041
				45,000	160	37	38.6	.110						
				40,000	439	42	39.8	.041						
84	4	4	2	53,000	79	29	25.4	———	50,500	39,500	29	37,000	48,500	.020
				50,000	109	29	27.5	.136						
				45,000	193	21	22.6	.057						
				40,000	360	28	28.2	.020						

[a] Square, forged as square bar; round, forged as round bar; all heats higher than 22 were forged as round bars only.
[b] Estimated.

TABLE XI.—COMPARISON OF 1200° F RUPTURE PROPERTIES OF WROUGHT HEAT-RESISTING ALLOYS

Alloys	Variable	Remarks	100-hr rupture strength (psi)	100-hr rupture elongation (percent)	Reference[a]
Modified alloys.	Chemical composition.	Prior processing and heat treatment controlled and held constant.	26,000 to 52,000	5 to 40	[b]
Basic alloy.	Heat treatment and hot-cold-work.	Single commercial heat.	40,000 to 66,000	1 to 50	1
Six standard heat-resisting alloys: 18Cr–8Ni. 18Cr–8Ni–1Cb. 16Cr–13Ni–3Mo. 25Cr–12Ni. 25Cr–20Ni. 15Cr–35Ni.	Chemical composition and prior processing.	Treatment: 2000° F, water-quenched. 2250° F, water-quenched. 2000° F, water-quenched. 2200° F, water-quenched. 2150° F, water-quenched. 2000° F, water-quenched.	 20,000 30,000 31,000 24,500 24,000 22,000	 20 8 15 7 7 3	4
Seven heat-resisting super alloys: 19–9DL. 17W. OSA. Timken. Low-carbon N–155. High-carbon N–155. S590.	Chemical composition and prior processing.	Treatment variable:[c] Ann, HW, CW. Ann, HW, CW. Ann, HW, CW. Ann, HW, CW. Ann, Ann+A, HW, CW. Ann, HW, CW. Ann, Ann+A.	 42,000 to 62,000 30,000 to 45,000 47,500 to 53,000 49,000 to 57,000 42,500 to 61,000 52,000 to 63,000 51,000 to 53,000	 2 to 20 2 to 24 5 to 25 9 to 28 5 to 17 4 to 11 6 to 20	5

[a] Numbers refer to references listed at end of text.
[b] Data from this investigation.
[c] Treatment variables: Ann—annealed (solution-treated).
Ann+A—solution-treated plus aged.
HW—hot-worked.
CW—hot-cold-worked.